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Friction, Wear, and Evaporation Rates of Various Materials in Vacuum to 10^{-7} mm Hg

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Evaporation data on soft metals, lubricating inorganic compounds, and various reference materials are reported for temperatures from 75 to 1000 F in vacuum as low as 10^{-7} mm Hg. Observations on modes of vacuum degradation (e.g., evaporation or dissociation) and methods of experimentation are related. Friction and wear data are presented for several unlubricated metals (e.g., type 440-C steel) and metals coated with inorganic (e.g., MoS_2 , CaF_2) as well as with soft metal films in vacuum at ambient pressures between 10^{-6} and 10^{-7} mm Hg.

Introduction

THE requirements for bearings and seals to operate in the environment of space dictate a new area for lubrication research. The low ambient pressures encountered in space can be expected to influence the behavior of oil, grease, and solid-film lubricants. The property of these materials most significantly affected by low ambient pressures is the evaporation rate. Various investigators have therefore measured the evaporation rates of oils and greases in vacuum as one method of establishing their relative merit for space applications (1-3). The results of this work have given some indication as to the oils and greases with the greatest stability at reduced ambient pressures. Only limited experimental work, however, has been reported in the literature for inorganic solids and soft metals which have potential use as solid lubricant films or coatings for hard alloy substrates [e.g. Reference (4)]. In general, the evaporation rates of these materials would be lower than those of oils and greases. These films might therefore be very attractive as lubricants for high vacuum service.

The lack of oxygen in outer space creates another problem for lubrication systems, namely, the absence of the protective metal surface oxides normally encountered in air. In the absence of these or other protective surface films, mass metal transfer, welding, and high coefficients of friction may be experienced for metals in sliding contact (5-7). Some data are presented in the literature for the friction and wear of metals and soft alloys in vacuum [e.g. Reference (8)]. In general, these data were obtained with conventional oil diffusion pumps

in the vacuum systems. One of the problems associated with such systems is that of back-migration of oil vapors from the pump to the test chamber. This back-migration even with cold traps and baffles can serve as a source of specimen contamination and result in a pronounced reduction in friction and wear.

There is a need for data on the evaporation rates of lubricants and also on the friction and wear characteristics of potential bearing and seal materials; these data must be obtained in a vacuum environment to facilitate the proper selection of lubricants and slider materials intended for use in space. The friction and wear data should be obtained in systems which are free from sources of specimen contamination like that associated with the oil diffusion pumps.

The objectives of this investigation were to determine in vacuum (10^{-6} to 10^{-7} mm Hg): (a) the evaporation rates for various organic and inorganic lubricants, (b) the evaporation rates of solid lubricant coatings, (c) the friction and wear properties of unlubricated slider materials for reference, and (d) the friction and wear properties of these slider materials coated with solid lubricant films. Evaporation-rate experiments were used to select the most promising solid lubricants for use as surface films on slider materials in vacuum. Evaporation rates were measured at temperatures from 55 to 1000 F. Friction and wear experiments were conducted with a 3/16-in. radius rider sliding on a 2½ in. diameter disk specimen at a surface speed of 390 ft/min. The rider was loaded against the disk with a 1000 g load. The duration of the experiments was 1 hr.

Materials

The specimens used in the evaporation experiments of this investigation were oils, greases, inorganic solids, metals, and solid lubricant coatings. The oils included MIL-L-7808 (di-2-ethyl hexyl sebacate), two mineral oils (viscosities of 380 and 235 centistokes at 100 F) designed for high-temperature use, and a polyphenyl ether [1-(p-α-cumyl phenoxy)-4-phenoxy benzene]. The ether was a solid at room temperature. The greases

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TABLE 1
Alloy Compositions

Metal	Composition												Hardness, Rockwell
	Fe	Ni	Co	Cr	C	Mo	W	Ti	Si	Mn	Al	Other	
52100	Bal. of comp.			1.3 to 1.6	0.95 to 1.10				0.20 to .35	0.25 to .45		P, 0.025 S, 0.025	R _c 60-43
440-C	Bal. of comp.			16.0 to 18.0	0.95 to 1.20	0.75			1.0 Max.	1.0		P, S	R _c 54
Ni-Cr	0.250	Bal. of comp.	11.0 to 12.0	18.0 to 20.0	0.06 to .12	9.0 to 10.5		3.2	0.010	0.050	1.50		R _c 43
Ni-Cr-Fe	5.0 to 9.0	70.0		15.0	0.08				0.50			S, Cb Al, Mn	R _c 29
Cobalt alloy	3.0	2.5	43.0	31.0	2.4		17.0		1.0				R _c 52-63
Ni-bonded TiC and CbC ^a		25.0			13.2	5.0		52.0				Cb, 4.5; Ta, 0.3	R _A 89

^a Ti and Cb are present as mixed carbides.

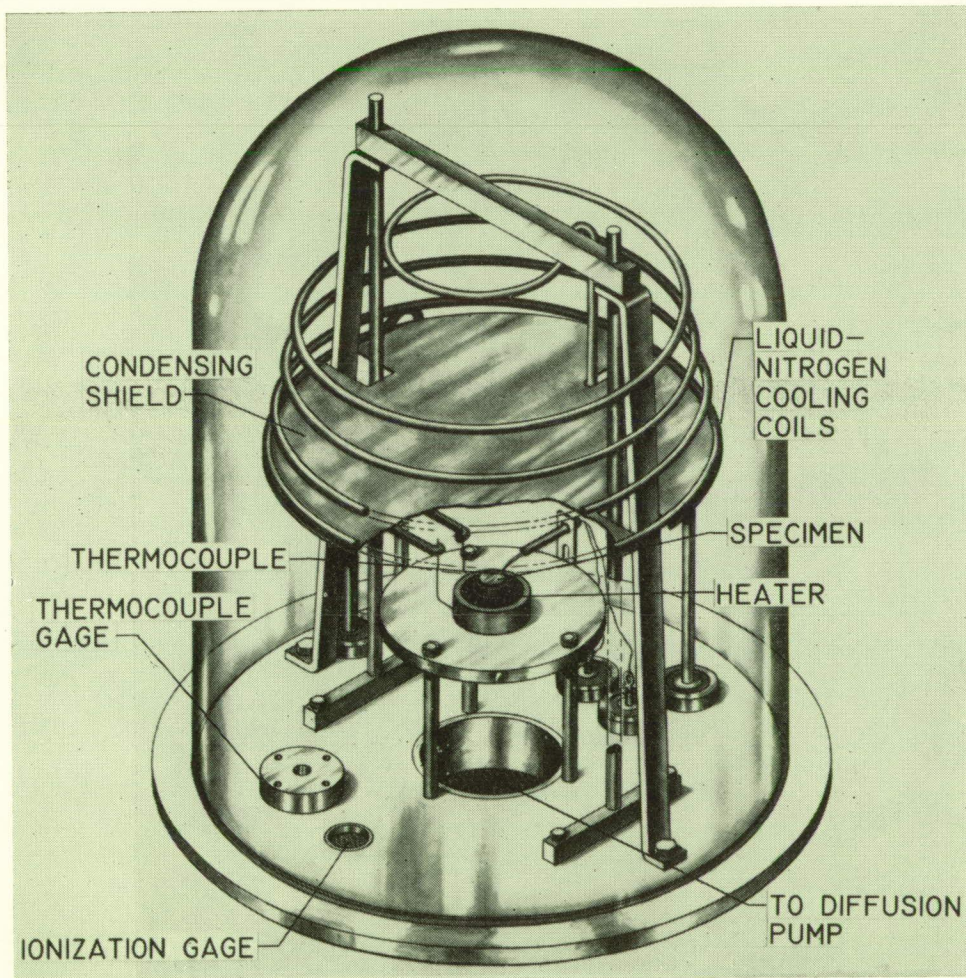


FIG. 1(a). Vacuum apparatus for bulk evaporation studies

included were a radiation-resistant grease and two mineral-oil-base greases with phthalocyanine thickeners. The inorganics included chemically pure materials obtained from commercial supply houses: cadmium iodide (CdI_2), cobalt chloride (CoCl_2), nickel fluoride (NiF_2), lead oxide (PbO), calcium fluoride (CaF_2), barium fluoride (BaF_2), tungsten disulfide (WS_2), and molybdenum disulfide (MoS_2). The nickel bromide (NiBr_2) could not be readily obtained commercially and was therefore prepared in the laboratory. The metals included were cadmium (Cd), indium (In), zinc (Zn), magnesium (Mg), tin (Sn), gallium (Ga), lead (Pb), and silver (Ag). Two organics other than the oils and greases for which evaporation rates were measured were polytetrafluoroethylene (PTFE) and epoxy (diglycidyl ether of bisphenol A with a diethylene triamine catalyst).

The alloys used in the friction and wear experiment with their compositions are presented in Table 1.

The molybdenum disulfide coatings used were coatings of the following types: silicon resin bonded, epoxy-phenolic resin bonded, ceramic bonded, and a metal matrix coating. The coatings were applied to 0.003 in. thick metal tabs for evaporation experiments. The thickness of the applied coating was 0.001 in. In the friction and wear experiments, however, the coating thickness on the disk specimens was 0.0002 to 0.0003 in.

Apparatus

The apparatus used in this investigation is presented in Figs. 1(a) to 1(c). The evaporation apparatus consisted of a commercial bell-jar vacuum system with a 4 in. oil diffusion pump system. Pressures in the 18 in. diameter chamber were measured with thermocouple and hot cathode ionization gages. Inside the bell-jar chamber just above the throat of the diffusion pump,

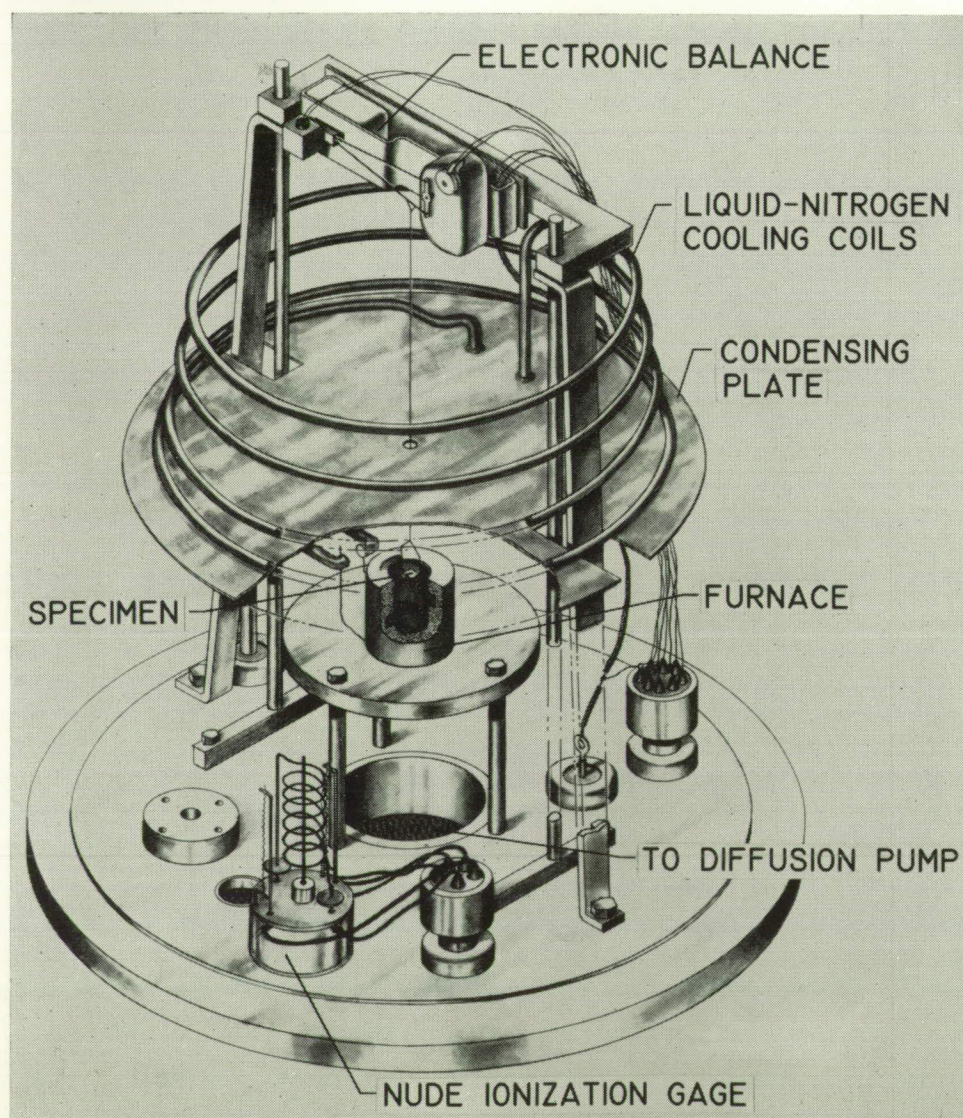


FIG. 1(b). Vacuum evaporation apparatus with balance for continuous rate measurements

a large plate was used to support a heater and the test specimen. The heater consisted of a housing of machinable ceramic and a wire-wound tungsten heating element. The specimen was placed on the heating element. A thermocouple was positioned on the top edge of the specimen for temperature measurement and control.

Approximately 4 in. above the specimen was a condensing shield. This shield was cooled with a liquid-nitrogen coil, and the temperature measured at the center of the condensing shield when liquid passed through the coil was -204°F . On the side of the shield facing the specimen was a copper tab for making X-ray analyses of condensate composition.

The bell-jar evaporation apparatus was modified for use with a continuous recording balance. The apparatus with associated modifications is presented in Fig. 1(b). The weighing mechanism of a commercially available electronic balance was mounted on a support in the upper part of the bell jar. Suspended from the balance beam was a thin wire that extended from the balance beam through the condensing shield to a stirrup that supported the specimen. The specimen and stirrup were suspended in the mouth of a cylindrical furnace. The walls of the furnace contained the heating elements of wound tungsten wire. A dummy specimen with thermocouple placed beneath the test specimen was used to

measure and control specimen temperatures. The dummy specimen technique has been employed by others in similar experiments (9). Weight changes in the specimen were continuously recorded on a strip chart.

The vacuum friction and wear apparatus is shown schematically in Fig. 1(c). The basic elements of the apparatus were a $2\frac{1}{2}$ in. diameter disk and a $3/16$ in. radius rider specimen. The disk was rotated by means of an induction motor with a canned rotor and stator. The enclosure of rotor and stator permitted a vacuum in the motor chamber. A vacuum valve connected the motor chamber with a mechanical pump system. The system included a liquid-nitrogen cold trap to inhibit oil migration from the pump. The specimen chamber containing the disk and rider was attached to the motor. The motor drive shaft extended into the specimen chamber with the disk specimen bolted directly to the end. The only seal between chambers was that afforded by a ball bearing mounted on the drive shaft. Since the presence of the ball bearing did not provide a vacuum seal, the ionization pump pumped both chambers. When the pressure in the specimen chamber was 10^{-7} mm Hg, it was approximately 10^{-4} mm Hg in the motor chamber.

The rider specimen was supported in the specimen chamber by a retaining arm that was gimbal and bellows mounted to the chamber. A linkage at the end of the

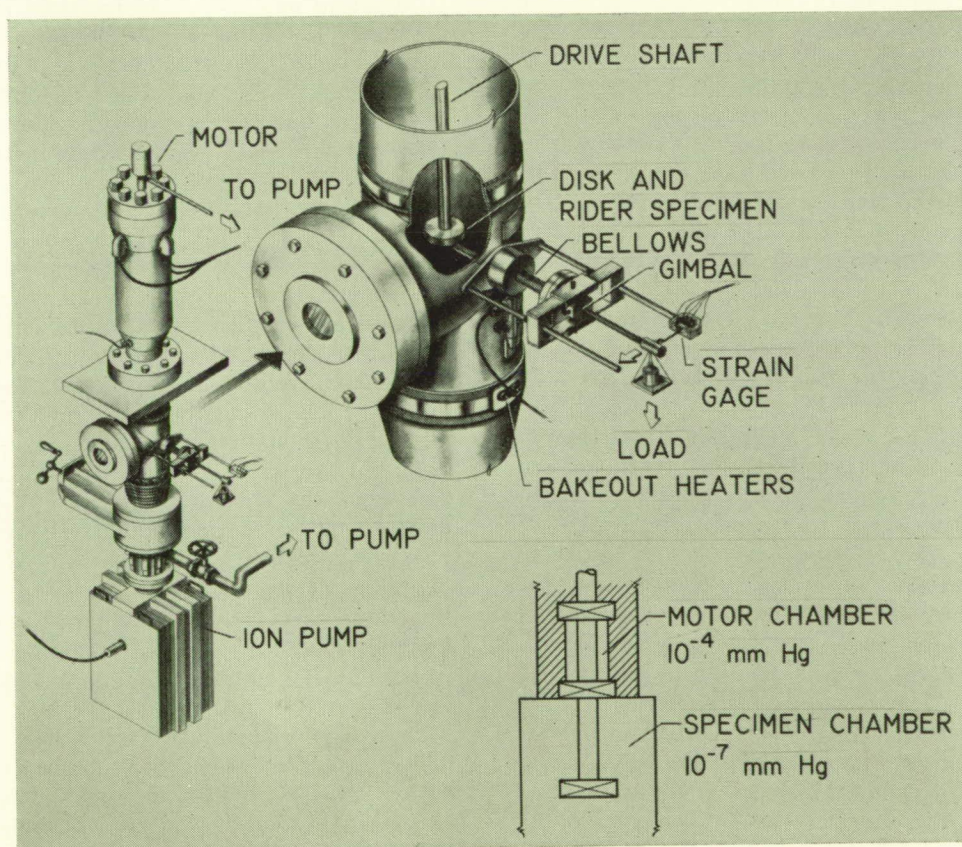


FIG. 1(c). Vacuum friction and wear apparatus

retaining arm away from the rider specimen was connected to a strain-gage assembly. The assembly was used to measure frictional force. Load was applied through a dead-weight loading system.

Attached to the lower end of the specimen chamber was a vacuum valve. The side of the valve away from the chamber had a T-section. On the lower portion of the T was connected a 400 liters/second ionization pump. The remaining portion of the T connected with a vacuum valve and a mechanical pump using liquid-nitrogen cold traps.

The first high-vacuum pump to be used with the vacuum friction and wear apparatus was a conventional oil diffusion pump. In initial experiments it was established, however, that specimen contamination by oil vapors had occurred (see Appendix A). Various liquid-nitrogen cold-trap designs were employed to eliminate this problem. Subsequent specimen examinations, however, indicated surface contamination. Combinations of cold traps, optical baffles, and heating grids for decomposition of oil vapors were then tried with little or no success. Since, even with the best trapping techniques, the possibility of specimen contamination did exist, the diffusion pump was replaced by an ionization pump. This pump employs no fluids or vapors in its operation.

Experimental procedure

The procedure used in the evaporation studies consisted of first preparing the experimental specimens. This was accomplished for the solids by oven-drying and dehydrating the materials. When the powders were dry, small charges were placed in a mold and compressed into tablet form under a 10,000 psi compression force. When the specimens were removed, they measured $\frac{5}{8}$ in. in diameter and were approximately $\frac{1}{8}$ in. thick. The tablets were then placed into a small preweighed glazed ceramic dish that had side walls of $\frac{1}{8}$ in. and inside diameter just large enough to accommodate the tablet specimen. The specimens and holders were then stored in a desiccator until ready for use. The oils and greases were placed directly in the dishes with the specimen filling the dish.

Prior to an experiment the specimens were removed from the desiccator and weighed. The specimen and holder were then placed into the vacuum chamber, and the chamber was evacuated. At a pressure between 10^{-6} and 10^{-7} mm Hg the experiment was started. In those experiments conducted above room temperature, the specimens were heated by a tungsten heater on which the specimen was placed.

The evaporation-rate experiments made with solid lubricant coatings were run using the electronic balance with a continuous measure of weight change with time. During an experiment a tab was placed on the stirrup that was suspended from the balance at the top end of the furnace, and the chamber was then evacuated. At a pressure between 10^{-6} and 10^{-7} mm Hg the evaporation experiments were started.

The disk and rider specimens used in friction and wear experiments were finish-ground at 4 to 8 μ in. Before each experiment the disk and rider were given the same preparatory treatment. This treatment consisted of (a) a thorough rinsing with acetone to remove oil and grease, (b) polishing with moist levigated alumina on a soft cloth, and (c) a thorough rinsing with tap water followed by distilled water. For each experiment (data point) a new set of specimens was used. In those experiments in which disks with coatings were used, the coatings were applied after the cleaning procedure. Plated specimens were solvent-cleaned, and bonded coatings were carefully handled following coating in order to avoid contamination.

The specimens were then placed in the apparatus. Mechanical pumps with liquid-nitrogen cold traps were used to obtain a pressure of 10^{-4} mm Hg in the specimen chamber. In the startup of the ionization pump, ionization of chamber gases was produced by the pump. This ionization generally persisted for 20 to 30 min. In the event that oil molecules had migrated through the liquid-nitrogen cold traps of the mechanical pumps prior to being valved off from the specimen chamber, the ionization would decompose them.

When the specimen chamber pressure was between 5.0×10^{-7} and 2.0×10^{-6} mm Hg as measured by a hot cathode ionization gage and by the ionization pump current, the friction experiment was started. A 1000 g load was applied to the rider loading it against the disk. Friction force was continuously measured and recorded on a strip chart. The wear was measured as volume loss of the rider specimen upon completion of 1 hr of running. The wear to the disk specimen surface was recorded with a surface profile measuring device.

Results and discussion

EVAPORATION DATA

The evaporation rates for some low-vapor-pressure oils and greases were determined in vacuum at various temperatures. The results obtained in these evaporation experiments are presented in Fig. 2. The sebacate MIL-L-7808 bubbled continuously while evaporating. The high-temperature mineral oils (viscosities of 380 and 235 centistokes at 100 F) did not exhibit this characteristic, and the surfaces were rather quiescent during evaporation at 55 and 200 F. The ether (1- α -cumyl phenoxy-4-phenoxy-benzene) was a solid at 55 F and exhibited no detectable weight change in 60.0 hr. At 200 F the ether was in a liquid state, and the evaporation rate was quite high (2.5×10^{-6} g/cm²/sec).

The greases appeared to lose the base oil with increase in temperature, leaving the bulk of the thickener behind. The evaporation experiments were terminated before complete evaporation of the base oil had occurred. This was found desirable in order to avoid a change in evaporation rates once the base stock had completely evaporated and the thickener with its own characteristic rate began to evaporate. The radiation-

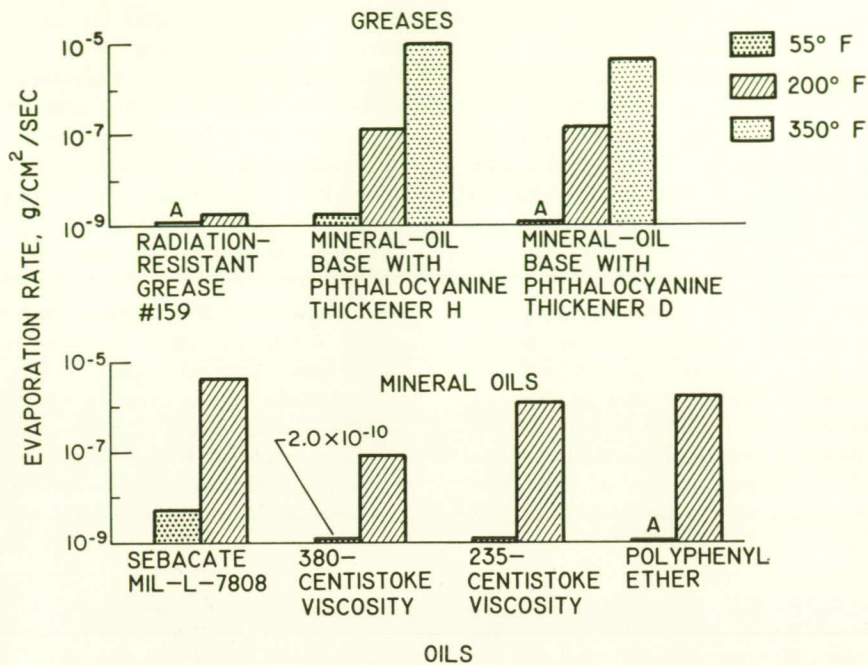
A - EVAPORATION RATE LESS THAN 1.0×10^{-10} g/cm²/SEC

FIG. 2. Evaporation rates for various oils and greases in vacuum. Ambient pressure, 8.0×10^{-7} to 2.0×10^{-6} mm Hg.

resistant grease exhibited a very low rate of evaporation at 55 F. At 200 F, however, the rate increased and the specimen became rubbery. The greases using phthalocyanine as a thickener both showed evidence of the thickener evaporating with the base stock, as determined by examination of the condensate on the liquid-nitrogen condensing shield. The evaporation rates of the greases were tolerable at 200 F; however, at 350 F the rate was quite high. The total per cent of the oil and grease samples lost depended upon the evaporation rates of the various materials. For example at 200 F only 3 per cent of the grease with H thickener was lost in 20 hr while 86 per cent of the MIL-L-7808 had evaporated in 14.0 hr. The oil and grease evaporation experiments were generally of 20 hr duration. In those cases where the evaporation rates were very high (MIL-L-7808) the experiments were terminated in shorter periods. Duplicate experiments indicated a reproducibility of data within 5 per cent.

The evaporation rates for compressed disks of various lubricant coating constituents were determined, and the results obtained in vacuum at various temperatures are presented in Fig. 3. The nickel fluoride showed no evidence of evaporation at 55 or 200 F. At 350 F, however, the nickel fluoride dissociated to metallic nickel and fluorine. The specimen surface changed from yellow-green to a black color. X-ray analysis of the surface indicated the black film to be metallic nickel. The lead oxide also dissociated at 350 F to metallic lead and oxygen. X-ray analysis indicated the presence of lead

on the specimen surface. The cobalt chloride sample exhibited a color change from blue to green at 500 F; however, the evaporation rate was low. X-ray analysis of the sample indicated its composition to be cobalt chloride (CoCl_2). The cobalt chloride, cadmium iodide, and nickel bromide evaporated and condensed on the condensing shield as cobalt chloride, cadmium iodide, and nickel bromide, respectively. The epoxy composition (diglycidyl ether of bisphenol A with a catalyst of diethylene triamine) showed evidence of decomposition at 350 F.

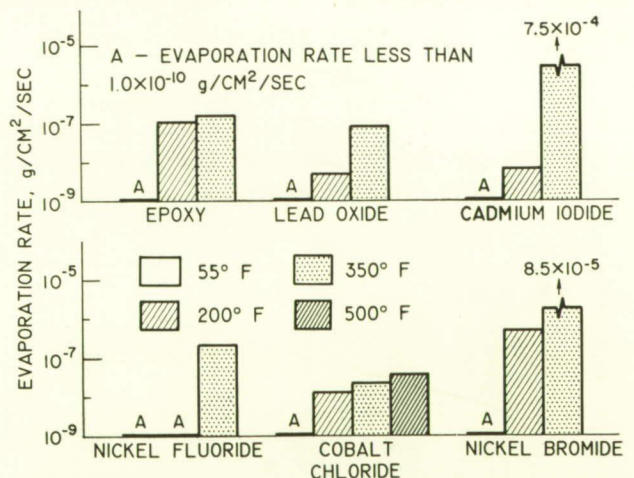


FIG. 3. Evaporation rates for possible coating constituents in vacuum. Ambient pressure, 8.0×10^{-7} to 2.0×10^{-6} mm Hg.

The evaporation rates for molybdenum disulfide, tungsten disulfide, calcium fluoride, and barium fluoride at temperatures to 1000 F are presented in Fig. 4. The weight losses for these materials in vacuum at elevated temperatures were very low, and in general these materials appeared to be the most stable of the inorganics tested. The rates for molybdenum disulfide, calcium fluoride, and barium fluoride were extremely close, and a single curve was drawn to represent the evaporation rate of these three materials. The molybdenum and tungsten disulfide had to be heated in vacuum prior to an experiment to remove adsorbed gases.

The evaporation rates for polytetrafluorethylene (pure polymer without plasticizer) were determined at various ambient temperatures and pressures. The results obtained in these experiments are presented in Fig. 5. While the polytetrafluorethylene exhibited some weight change at various ambient pressures, this weight change was extremely small. The evaporation rate of polytetrafluorethylene at various temperatures was determined at an ambient pressure of 8.0×10^{-7} to 2.0×10^{-6} mm Hg. The evaporation rate of polytetrafluorethylene was low to 350 F; at 650 F the rate was high. Polytetrafluorethylene begins to decompose at temperatures above

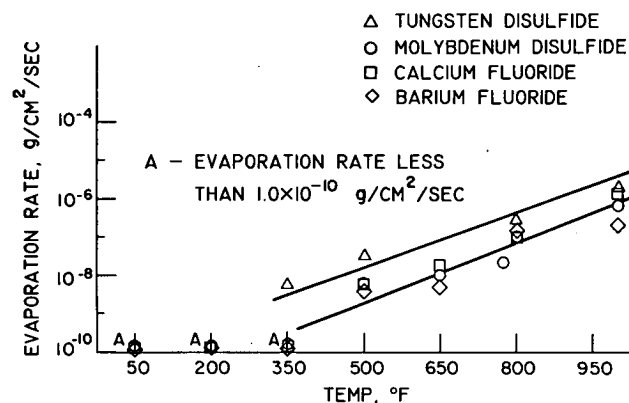


FIG. 4. Evaporation rates for various inorganic compounds in vacuum. Ambient pressure, 8.0×10^{-7} to 2.0×10^{-6} mm Hg.

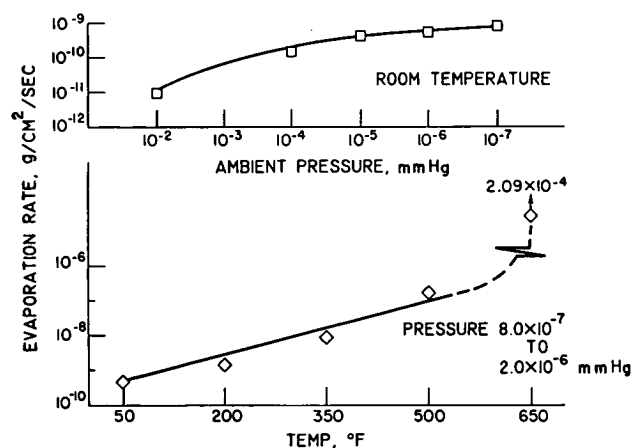


FIG. 5. Evaporation rate of PTFE at various temperatures and pressures.

500 F; this may well account for the high evaporation obtained at 650 F.

The evaporation rates for various metals were determined in vacuum over a range of temperatures, and the results obtained in these experiments are presented in Figs. 6 and 7. Cadmium and zinc exhibited relatively high rates of evaporation. The rates were higher than obtained with polytetrafluorethylene. Lead, tin, and silver had the lowest rates of evaporation of the soft metals investigated.

When the evaporation rates determined experimentally in this investigation are compared with data calculated from vapor pressure presented in the literature (10), the results appear to be in good agreement (Fig. 6). Any differences observed could be due not only to experimental error but to error in the calculated values. The calculated values are based on thermodynamic relations where an error of 10 per cent is not unlikely. Since the evaporation rate can be determined from vapor pressure, the reverse must also be possible. The use of evaporation

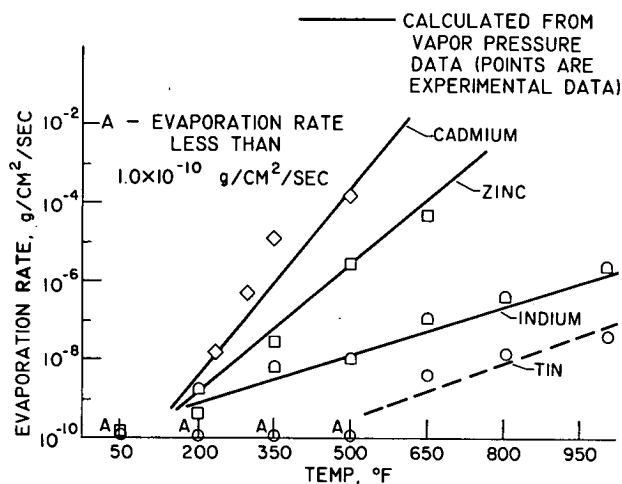


FIG. 6. Evaporation rates for various metals in vacuum. Ambient pressure, 8.0×10^{-7} to 2.0×10^{-6} mm Hg.

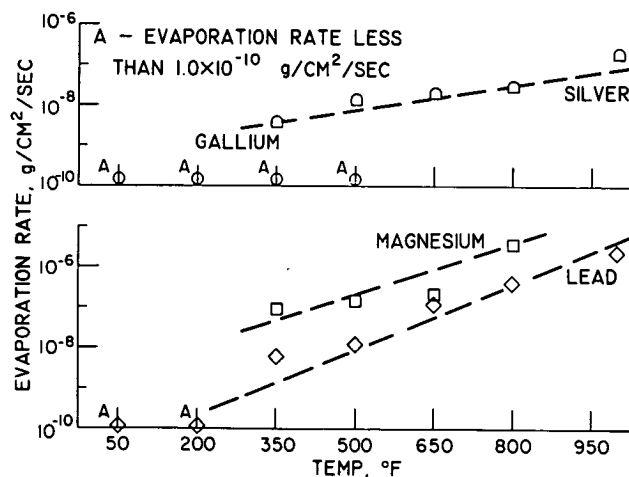


FIG. 7. Evaporation rates for various metals in vacuum. Ambient pressure, 8.0×10^{-7} to 2.0×10^{-6} mm Hg.

rates to determine vapor pressure can be accomplished for pure metals by a simple equation:

$$p_{\text{mm}} = 17.14 G \sqrt{\frac{T}{m}}$$

where

- p_{mm} = vapor pressure in mm Hg
 G = evaporation rate in g/cm²/sec
 T = temperature in °K
 m = molecular weight

The vapor pressure of zinc is determined from its evaporation rate at 500 F:

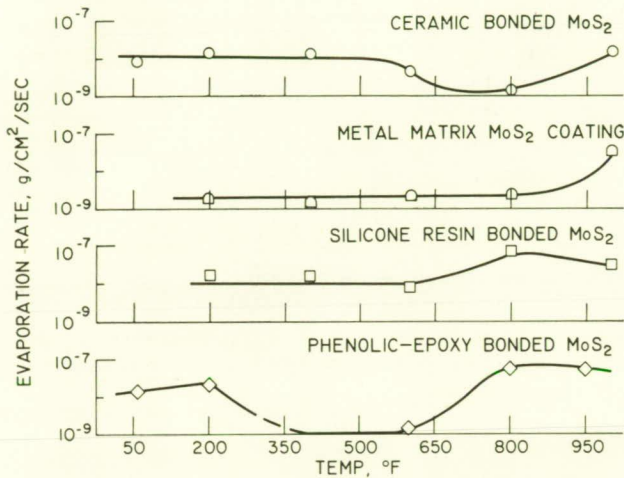


FIG. 8. Evaporation rate of various MoS₂ coatings in vacuum. Ambient pressure, $1.0\text{--}2.0 \times 10^{-6}$ mm Hg; 0.001-in. MoS₂ coating on Ni-Cr alloy.

$$p_{\text{mm}} = 17.14 (2.0 \times 10^{-6}) \sqrt{\frac{533}{65.38}}$$

$$p_{\text{mm}} = 9.81 \times 10^{-5}$$

The vapor pressure of zinc at 500 F presented in the literature (10) is 1.0×10^{-4} mm Hg.

Evaporation-rate experiments were conducted with four molybdenum disulfide coatings in vacuum at various temperatures. An electronic balance was used to continuously measure weight changes with time. The results obtained in these experiments are presented in Fig. 8. In general, there appears to be a break in evaporation

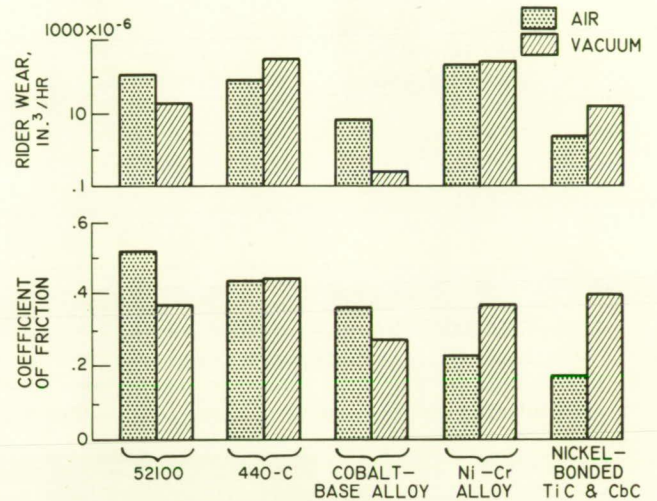


FIG. 9. Friction and wear of various alloys in air and in vacuum. Vacuum, 5.0×10^{-7} to 2.0×10^{-6} mm Hg; sliding velocity, 390 ft/min; load, 1000 g; duration of run, 1 hr.

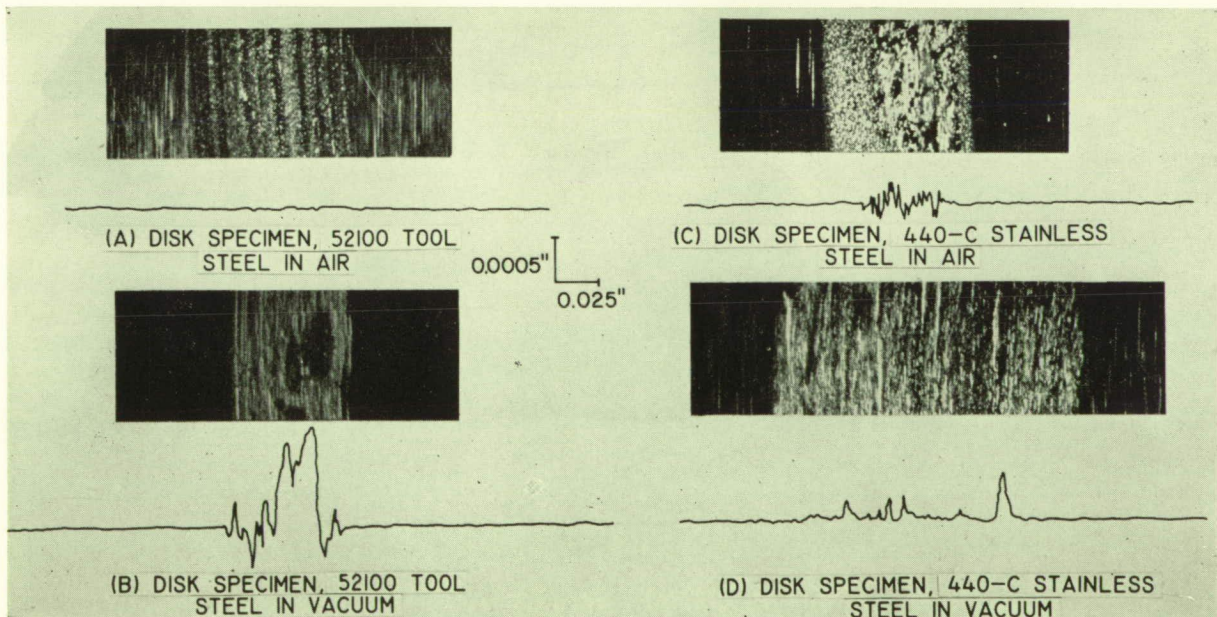


FIG. 10(A)–(D). Photomicrographs and surface profile tracings of wear areas on disk specimens, load, 1000 g; sliding velocity, 390 ft/min; duration of run, 1 hr; $\times 20$. (A) Disk specimen, 52100 tool steel in air. (B) Disk specimen, 52100 tool steel in vacuum. (C) Disk specimen, 440-C stainless steel in air. (D) Disk specimen, 440-C stainless steel in vacuum.

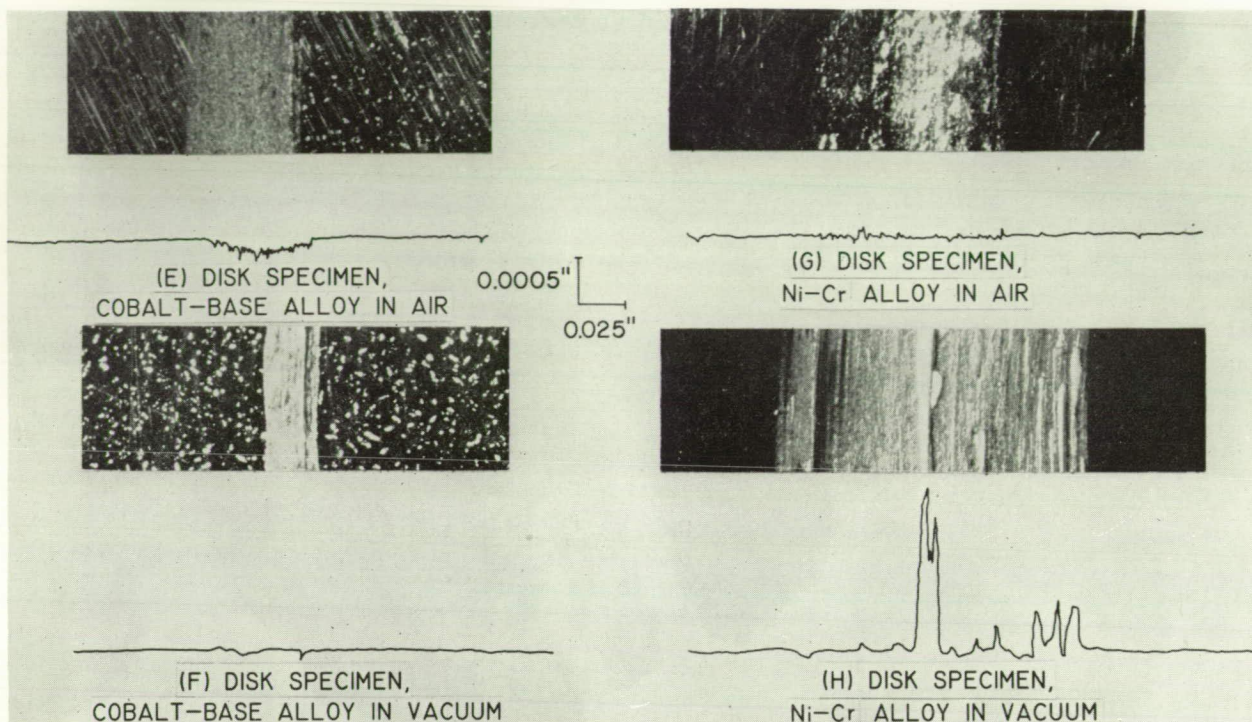


FIG. 10(E)-(H). Photomicrographs and surface profile tracings of wear areas on disk specimens, load, g; sliding velocity, 390 fpm; duration of run, 1 hr; $\times 20$. (E) Disk specimen, cobalt-base alloy in air. (F) Disk specimen, cobalt-base alloy in vacuum. (G) Disk specimen, Ni-Cr-Al alloy in air. (H) Disk specimen, Ni-Cr-Al alloy in vacuum.

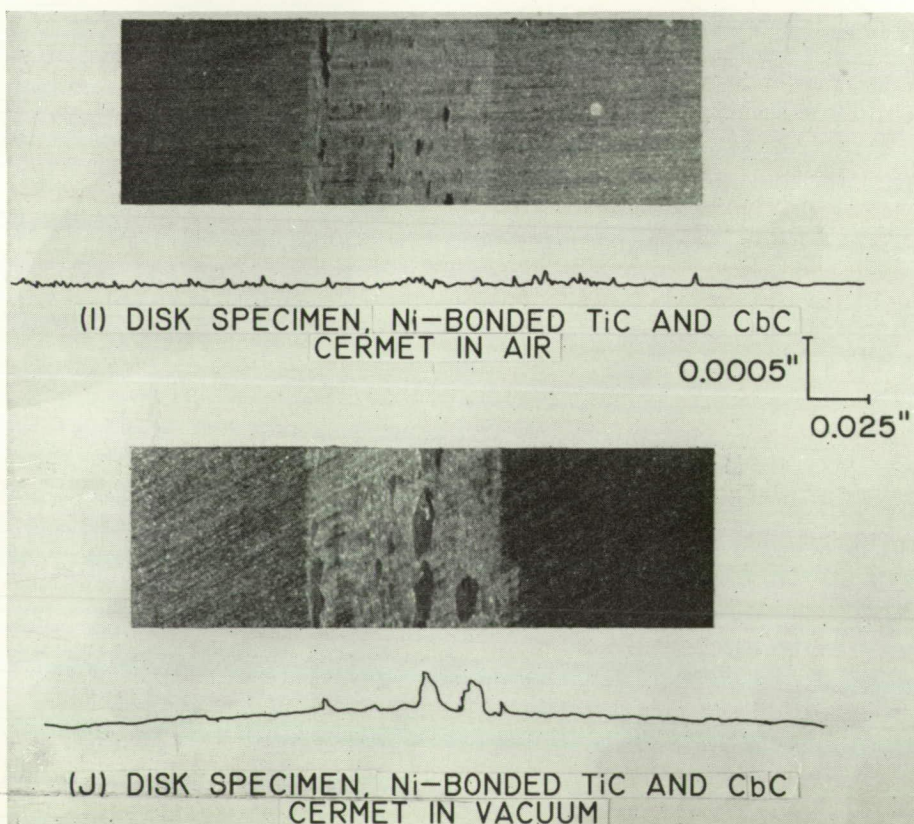


FIG. 10(I), (J). Photomicrographs and surface profile tracings of wear areas on disk specimens, load, g; sliding velocity, 390 fpm; duration of run, 1 hr; $\times 20$. (I) Specimen and rider, Ni-bonded TiC and CbC cermet in air. (J) Specimen and rider, Ni-bonded TiC and CbC cermet in vacuum.

curves for all coatings; this result indicates a change in evaporation rate. This change may be due to two different evaporation rates, one for the binder, and the other for molybdenum disulfide itself.

FRICITION AND WEAR DATA

Unlubricated metals. Friction and wear experiments were conducted in air and vacuum with five alloy combinations. The results obtained in these experiments are presented in Fig. 9. The friction and wear values for 52100 steel sliding on itself were lower in vacuum than in air. Examination of the disk surfaces after the experiments indicated a change in the wear mechanism [Figs. 10(a) and (b)]. Free metal wear particles are oxidized in air, and the resulting metal oxides on the particles prevent adherence to the base metal. At an ambient pressure of 10^{-6} mm Hg, however, where oxygen availability was appreciably reduced, the wear surface of the disk showed evidence of considerable metal transfer as nascent wear particles readily adhered to the parent metal. The surface profile tracings of Fig. 10(b) indicate "buildup" on the disk surface. This was apparently due to the transfer of metal from the rider to the disk specimen.

The low friction coefficients experienced for 52100 steel in vacuum (10^{-6} mm Hg) may be accounted for in that, even at 10^{-6} mm Hg, oxygen is present. Bowden and Tabor (11) have indicated that copper will oxidize as rapidly at 10^{-3} mm Hg pressure as it will at atmospheric pressure; also, it has been established elsewhere that, at 10^{-6} mm Hg, a clean metal surface will adsorb a monolayer of gas in a second. Since some oxygen is available at 10^{-6} mm Hg, it is probable that as pieces of metal, which are quite hot, transfer from one specimen surface to the other, localized oxidation of the transferred metal occurs. With the limited oxygen available the lower oxides of iron, FeO and Fe_3O_4 , would

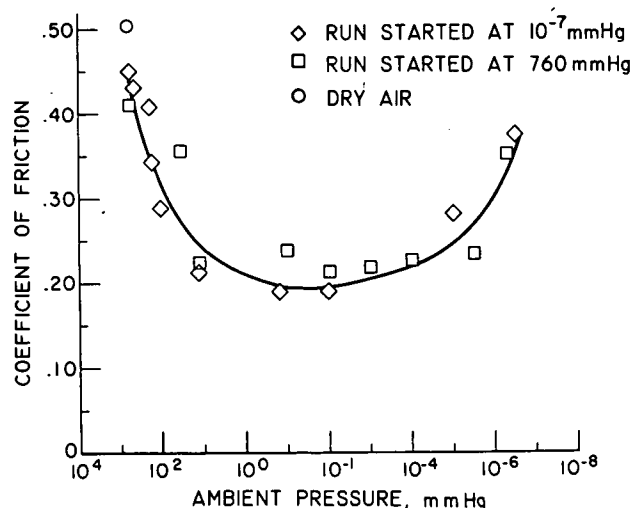


FIG. 11(a). Coefficient of friction for 52100 sliding on 52100 at various ambient pressures. Sliding velocity, 390 fpm; load, 1000 g; temperature, 75 F.

form on ferrous metal and result in a friction coefficient lower than normally encountered in air at atmospheric pressure (12) where Fe_2O_3 is one of the oxides present. Results obtained at pressures lower than 10^{-6} mm Hg should differ from those obtained at 10^{-6} mm Hg.

The coefficient of friction for 52100 tool steel sliding on 52100 tool steel at various ambient pressures is presented in Fig. 11(a). The friction decreased from 0.45 at 760 mm Hg pressure to 0.2 between 10^{-1} and 10^{-2} mm Hg. The friction began to increase again at 10^{-4} mm Hg, and at 5.0×10^{-7} mm Hg it was 0.375.

In order to reduce the oxygen concentration available for surface reaction at pressures of 10^{-6} to 10^{-7} mm Hg a liquid helium condensing coil was added to the friction and wear apparatus. Liquid helium will condense all gases except helium. A friction experiment was made at 2.0×10^{-7} mm Hg using the liquid helium condenser. The results obtained are presented in Fig. 11(b). The initial coefficient of friction was 0.2. This low value being associated with the presence of the lower iron oxides on the specimen surfaces. As time progressed, however, the coefficient of friction began to increase and after 30 min of operation when the residual surface oxides had worn away, a friction of 5.0 was observed. The specimen then welded together and the experiment was terminated by this complete seizure.

The friction and wear results obtained with 440-C stainless steel sliding on itself in vacuum were about the same as in air (Fig. 9). Examination of surface photomicrographs and profile tracings of the disk wear area [Figs. 10(c) and (d)] indicate that particles were transferred to the disk specimen.

The friction and wear of a cobalt-base alloy were determined in vacuum and air, and the results obtained are presented in Fig. 9. The friction and wear were lower in vacuum than in air, and examination of the surface topography indicated no evidence of mass metal transfer in vacuum [Fig. 10(f)]. Similar results have been observed for this particular alloy in inert and reducing

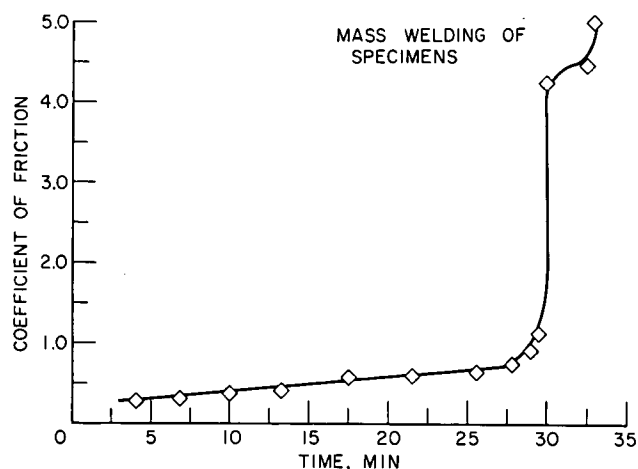


FIG. 11(b). Coefficient of friction for 52100 sliding on 52100 in vacuum with liquid helium cryogenic pumping; pressure 2.0×10^{-7} mm Hg, sliding velocity 390 fpm, load 1000 g.

atmospheres (data in process of publication). This particular alloy is an air cast material and has about 1.0 per cent silicon in its composition. The influence of silicon on the friction and wear properties of materials is discussed in Reference (11). It was established in (11) that small quantities of silicon rather markedly reduced the friction and wear normally encountered for alloys in inert and reducing atmospheres. This same mechanism could be expected to apply in vacuum.

The friction and wear properties of a vacuum-melted nickel-base alloy (Ni-Cr) were next determined. The results obtained in air and vacuum are presented in Fig. 9. The coefficient of friction in vacuum was greater than in air. The rider wear, however, was about the same in both environments. Examination of this wear area on the disk specimen indicated metal transfer in vacuum [Fig. 10(h)]. From the photomicrograph it appears that pieces of metal were transferred from rider to disk and then smeared in the direction of sliding. Though the rider wear was nearly the same in air and in vacuum, the wear mechanism on the disk surfaces was quite different, as indicated by comparison of the photomicrograph and surface profile traces of Figs. 10(g) and 10(h).

The friction and wear values for a nickel-bonded titanium and columbium carbide cermet sliding on itself is presented in Fig. 9. For this particular material both friction and wear were higher in vacuum than in air. The photomicrograph and surface profile tracing of Fig. 10(j) indicate evidence of metal transfer to the disk surface in vacuum.

Lubricated metals. The mechanism of wear for metals in vacuum indicates that stable films should be employed to lubricate these materials. Since molybdenum disulfide has been shown to be a good dry film lubricant in air and has very low evaporation rates in vacuum,

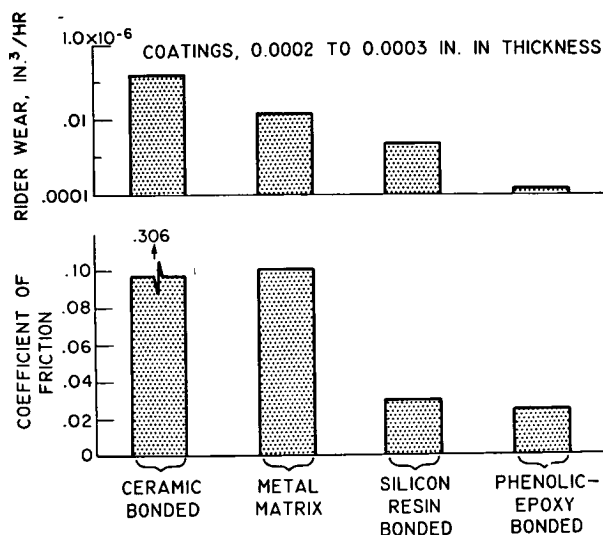


FIG. 12. Friction and wear of 440-C on 440-C with various MoS_2 films. Ambient pressure, 8.0×10^{-7} to 2.0×10^{-6} mm Hg; sliding velocity, 390 fpm; load, 1000 g; duration of run, 1 hr.

molybdenum disulfide coatings using various binders were used as solid-film lubricants for 440-C stainless steel in vacuum. The friction and wear results obtained with 440-C sliding on 440-C coated with various molybdenum disulfide films in vacuum are presented in Fig. 12. From the results obtained, it appears the lubricant binder plays an important role in the friction and wear results obtained with molybdenum disulfide coatings. The best friction and wear were obtained with an epoxy-phenolic and a silicone resin-bonded molybdenum disulfide coating. The metal matrix was less satisfactory, and the ceramic bonded gave the poorest results with a continuous friction coefficient of about 0.3 over an entire 1 hr of operating. These results, however, were obtained at room temperature, and the epoxy-phenolic coating may not appear as promising at elevated temperatures.

Friction and wear experiments were made with other solid film, and the results obtained are presented in Fig. 13. A lead oxide-silicon dioxide coating developed for high-temperature use in air was applied to 440-C stainless steel and was run in vacuum. A friction coefficient of about 0.17 was obtained at 10^{-6} to 10^{-7} mm Hg ambient pressure. In air at room temperature, with a sliding velocity of 430 ft/min, this particular coating had a friction coefficient of 0.3 (14). In vacuum the surface became sufficiently heated without an external heat addition to give a friction coefficient similar to that obtained in air at 250 F in (14).

A calcium fluoride solid lubricant coating developed for high-temperature applications was applied to a Ni-Cr disk. A Ni-Cr-Fe rider was used against the disk in vacuum. The friction and wear obtained are presented in Fig. 13. The coating gave a friction coefficient of 0.18 over a 1 hr period, and the wear to the rider specimen was low. The friction coefficient obtained with this coating in vacuum was lower than obtained in air at the same temperature.

In the literature, instances are cited where thin, soft metal films were used as protective coatings for bearings run in vacuum (4, 15). Various soft metals were there-

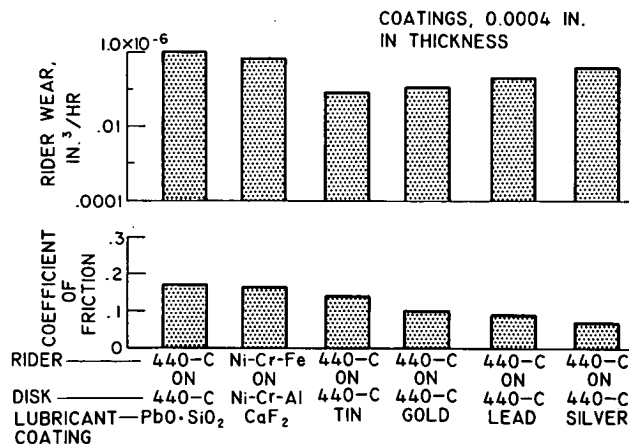


FIG. 13. Friction and wear of alloys with various coatings in vacuum. Ambient pressure, 8.0×10^{-7} to 2.0×10^{-6} mm Hg; sliding velocity, 390 fpm; load, 1000 g; duration of run, 1 hr.

fore applied to 440-C stainless steel substrates to determine their influence on the friction and wear of these substrates in vacuum. The results obtained with a 440-C stainless steel rider sliding on the thin metal film (0.0004 in. thickness) coated 440-C disks are presented in Fig. 13. The friction coefficient for lead, gold, and silver was less than 0.1 while tin was about 0.14. The wear for 440-C stainless steel was low with all four coatings. The thin metal films appear to have properties when applied to hard substrates that make them potential candidates as solid-film lubricants for use in vacuum.

Summary of results

Evaporation rates, and friction and wear were obtained at a pressure of 10^{-6} to 10^{-7} mm Hg. Since the pressures encountered in space may be many orders of magnitude lower (approximately 10^{-14} mm Hg), the results obtained in similar experiments at lower pressures may differ somewhat from those obtained herein. The results presented here, however, do give some indication of the effects of reduced ambient pressures on lubricants, friction, and wear. The results obtained in this study are summarized as follows:

1. The use of MoS_2 coatings (epoxy-phenolic resin and silicone resin bonded 0.0002 to 0.0003 in. thickness) provided effective solid lubricant films for 440-C stainless steel at pressures of 10^{-6} to 10^{-7} mm Hg. Both friction and wear were extremely low with these coatings.

2. Some metals (Ag, Sn, Au, and Pb) when applied to substrates such as 440-C stainless steel (0.0004 in. thickness) appreciably reduce the friction and wear normally encountered with this alloy in vacuum (10^{-6} to 10^{-7} mm Hg).

3. The friction and wear results obtained with various metals in vacuum (10^{-6} to 10^{-7} mm Hg) indicate a wear mechanism unlike that encountered in air. In general the wear in vacuum was characterized by mass metal transfer. The friction coefficients were lower than obtained in air for some alloys (e.g., 52100) and considerably higher for others (e.g., Ni-Cr alloy and Ni-bonded TiC and CbC). Operation at lower pressures might be expected to further alter the wear mechanism based on oxygen availability.

4. The evaporation data for some inorganics (CaF_2 and MoS_2) and metals (Ag and Sn) indicate they have potential for use as lubricants in vacuum (10^{-6} mm Hg) to temperatures as high as 1000 F.

5. The evaporation data indicate that some oils and greases may be used as lubricants at pressures of 10^{-6} mm Hg for short periods of time provided the ambient temperature is low (55 F).

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Appendix

A STUDY OF THE EFFECTIVENESS OF VARIOUS TRAPPING DEVICES IN STOPPING BACK-MIGRATION OF DIFFUSION PUMP OILS

The vacuum friction and wear apparatus used in this investigation originally employed an oil diffusion pump to obtain high vacuum. The oil diffusion pump has associated with it the undesirable characteristic of permitting oil vapors to migrate from the pump. In the vacuum literature it has been suggested that this problem can be eliminated by the use of baffles and/or cold traps between the oil diffusion pump and experimental

chambers. Numerous cold-trap and baffle designs have been reported in the literature and users of oil diffusion pumps have employed these and other modifications in vacuum systems. There appears, in general, to be very little if any standardization on cold-trap and baffle designs. Various cold traps and baffles were therefore tried in the vacuum friction and wear apparatus in an attempt to eliminate back-migration of oil vapors.

The first step taken was to place a liquid-nitrogen spiral cold trap in the apparatus between the experimental chamber and the oil diffusion pump. A schematic of this cold trap is shown in Fig. 14(a). The gas being

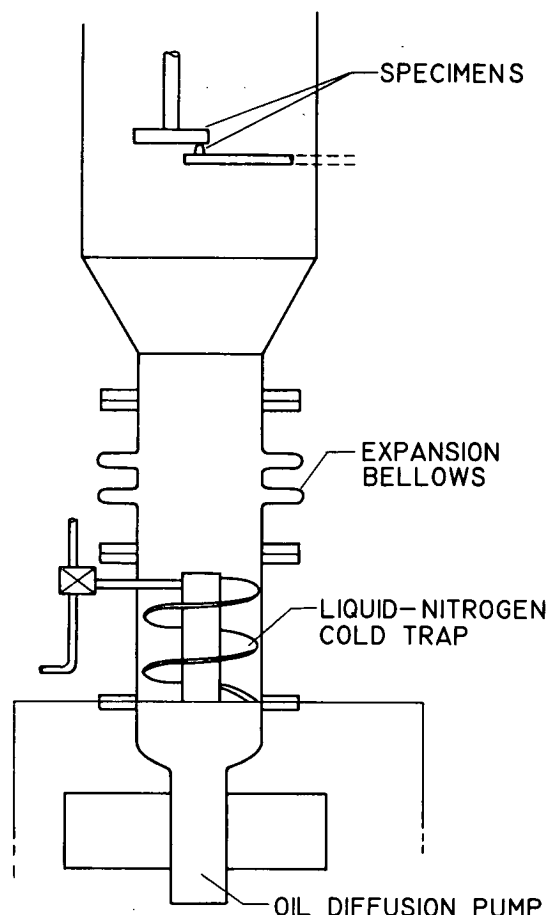


FIG. 14(a). Various baffle designs used in vacuum friction and wear apparatus.

pumped had to travel a spiral path along liquid-nitrogen-cooled walls. A friction experiment was conducted with a disk and rider specimen in the experimental chamber. The friction coefficient obtained with clean 440-C stainless steel was in a region (0.1) associated with effective boundary lubrication. The disk surface was checked with distilled water after the experiment; observations of high contact angle and lack of wettability gave evidence of an oil film.

An optical baffle plate was then added between the cold trap and specimens in an attempt to eliminate the contamination. The baffle design used is shown in Fig.

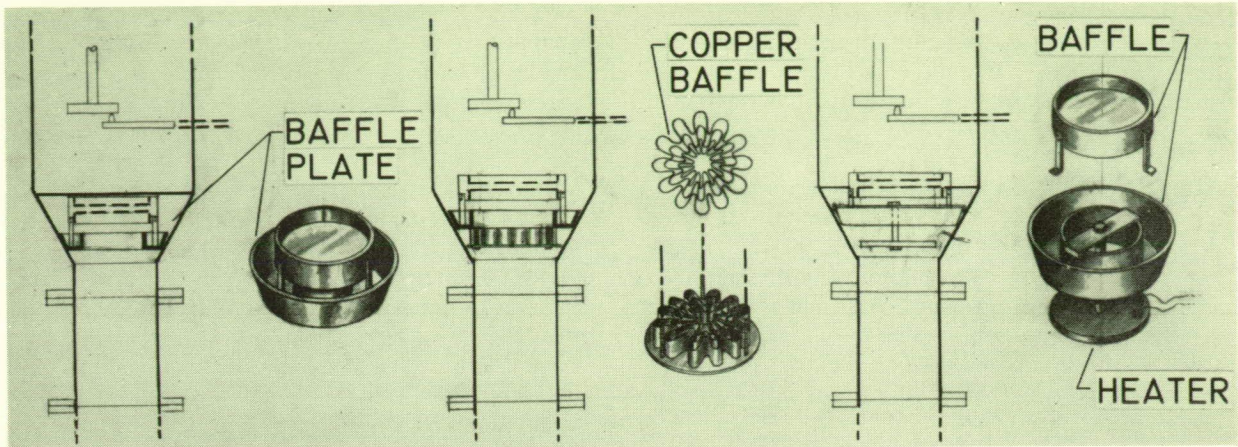


FIG. 14(b), (c), (d). Various baffle designs used in vacuum friction and wear apparatus

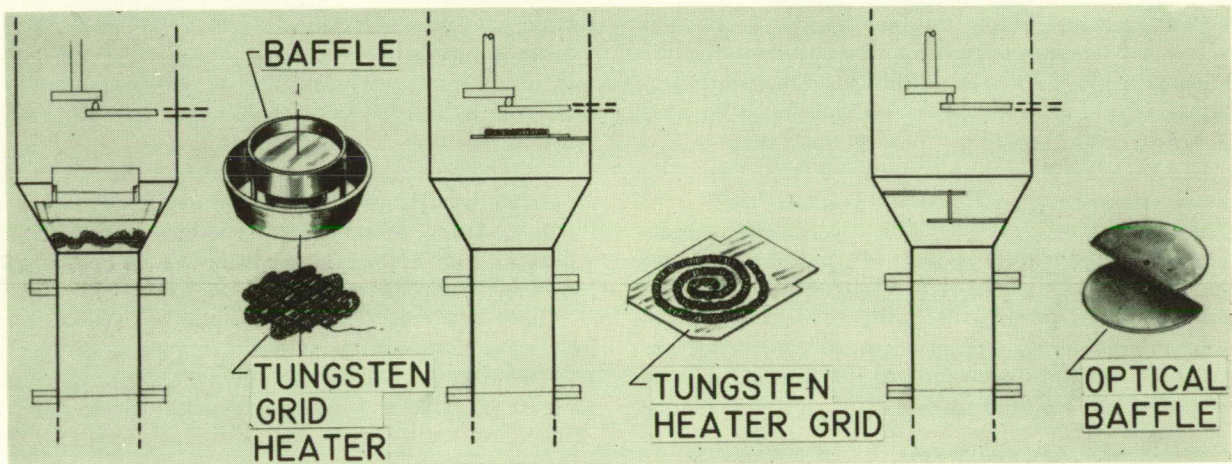


FIG. 14(e), (f), (g). Various baffle designs used in vacuum friction and wear apparatus

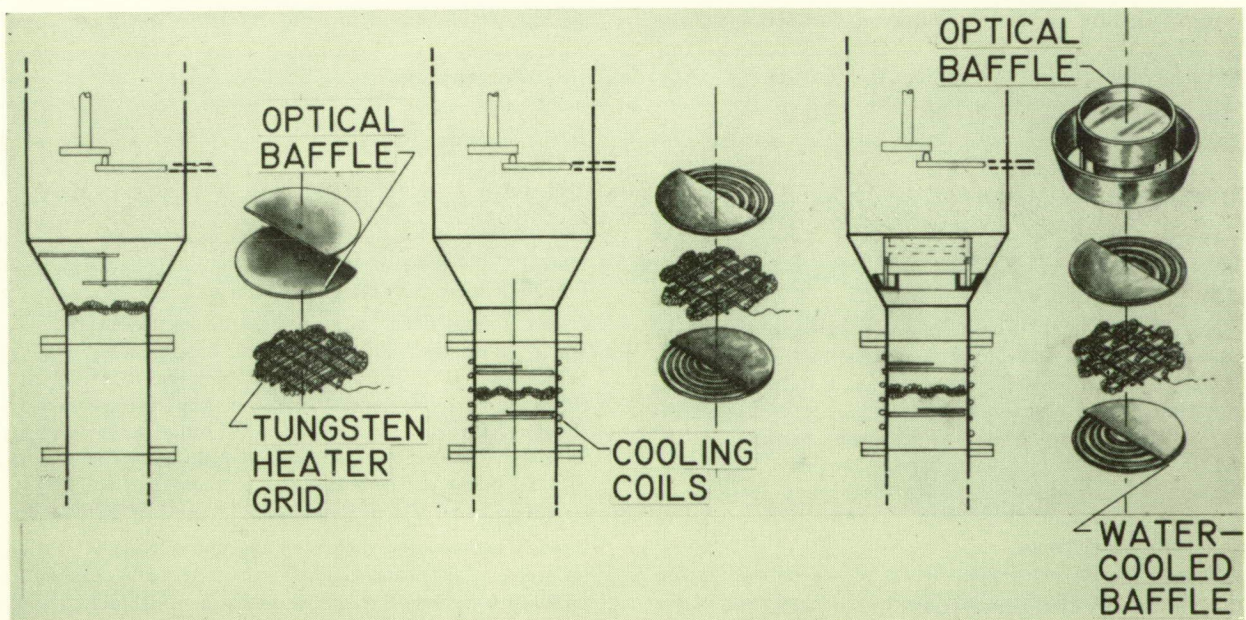


FIG. 14(h), (i), (j). Various baffle designs used in vacuum friction and wear apparatus

14(b). Friction experiments with 440-C stainless steel again gave a friction coefficient indicative of effective boundary lubrication. Examination of the disk surface with distilled water showed evidence of a contaminated surface.

The use of a honeycomb copper structure as a baffle has been reported in the literature as a means of trapping oil molecules. A baffle of this design was fitted to the optical baffle, and the unit was placed in the specimen chamber above the liquid-nitrogen cold trap [Fig. 14(c)]. This arrangement was not effective in preventing specimen contamination by oil vapors.

Another approach to eliminating the back-migration problem considered was that of using heat to decompose oil vapors migrating to the specimen chamber. To achieve this, a heater was attached to the optical baffle previously used. The optical baffle with heater placed between it and the liquid-nitrogen cold trap is shown in Fig. 14(d). This arrangement was also inadequate in eliminating specimen contamination, as ascertained by friction experiments with clean 440-C stainless steel and wettability checks. The heater plate was then replaced by a heater grid of tungsten coils. These coils were heated to an orange-yellow heat in an attempt to decompose oil vapors entering the specimen chamber. The heater grid and optical baffle used are shown in Fig. 14(e). This arrangement was also unsatisfactory.

A tungsten-wound heater coil was then placed in the specimen chamber directly beneath the disk and rider in an attempt to keep vapors from the specimen surfaces [Fig. 14(f)]. This arrangement was insufficient, and the specimens again exhibited evidence of surface contamination.

The design of the optical baffle previously used was altered, and the new optical baffle is shown in Fig. 14(g), where it was used in conjunction with the liquid-nitrogen cold trap. In Fig. 14(h) a tungsten heater grid was added below the baffle. Neither of these systems accomplished the elimination of oil contamination as deter-

mined by friction coefficients and specimen surface examination.

The next approach to the back-migration problem considered was to remove the expansion bellows of the apparatus located between the specimen chamber and the liquid-nitrogen cold trap and to replace it with a heater-baffle assembly. This assembly can be seen in Fig. 14(i). It consisted of two water-cooled optical baffle plates with a tungsten-wound heater grid between the plates. The temperature of the space between the tungsten grids maintained during diffusion pump operation was 1000 F. The outside walls of the chamber were water-cooled. This assembly in conjunction with the liquid-nitrogen cold trap minimized the specimen contamination. Examination of the disk surface with distilled water indicated a contact angle less than previously obtained; however, the water still would not wet the metal surface, and the coefficient of friction for 440-C stainless steel was about 0.2. An addition was then made to the system, namely, the optical baffle used in earlier trapping designs. The complete trapping system can be seen in Fig. 14(j). The results obtained were about the same as with the arrangement of Fig. 14(i).

A liquid-nitrogen-cooled plate type chevron baffle was also considered for these experiments. This configuration was not, however, used because experimental evidence by colleagues showed that it also allowed test-chamber contamination.

The experiences related here were obtained with a common diffusion pump oil composed of petroleum hydrocarbons. Separate experiments were conducted with additional diffusion pump oils including a silicone and a sebacate. In no case was back-migration of diffusion pump oil completely eliminated.

The inability to completely eliminate the back-migration of oil vapors from the oil diffusion pump resulted in the replacement of the oil diffusion pump with an ionization pump. This pump employs no fluid or vapors in its operation.

DISCUSSION

M. H. HABLANIAN AND E. G. JACKSON (*National Research Corporation, Cambridge 42, Massachusetts*):

We wish to comment particularly on the appendix of this paper. It is apparent from the difficulties the authors had with diffusion pumps that there is a possibility of oil contamination in diffusion pump systems. However, there is much evidence available indicating that the difficulty is not an inherent one, and can be avoided by suitable baffle design and pumpdown techniques. The authors' success with an ion-mechanical pump combination shows that backstreaming can be trapped even at pressures above the ultrahigh vacuum range.

The authors do not give many details of the diffusion pump system and pumping procedure, such as the size and type of the diffusion pump, the pump-down sequence and associated pressures, the presence or absence of separate roughing line, etc. It is

therefore difficult to determine exactly all possible conditions contributing to excessive backstreaming.

The obvious difficulty appears to be insufficient trapping. The major difficulties with the trapping arrangements used by the authors are: (1) The main liquid nitrogen cold trap does not ensure at least one contact on a cryogenic surface by molecules coming from any direction; (2) All other baffle arrangements used above the main liquid nitrogen trap are insufficient because they are either not cooled at all, or cooled only by water.

The spiral liquid nitrogen cold trap permits molecules to pass through between the spiral and the uncooled walls. In addition molecules which happen to travel along paths parallel to the spiral surface, bouncing off warm outer walls, will pass through the traps without contacting the cooled parts of the trap. After some of the oil molecules appear in the space above the cold trap,

it is useless to try to stop them from entering the working chamber by optical baffles if those baffles are not refrigerated.

It is important to distinguish between primary backstreaming which occurs in a well-defined pattern from the top jet of the pump, and the molecules re-evaporating from the walls which come from random directions and cannot be stopped by uncooled baffles. A well-designed liquid nitrogen baffle must assure at least one contact on a refrigerated surface for molecules coming from any direction. In sensitive experiments it may be desirable to have a minimum of two or more such contacts. N. Milleron at a recent American Vacuum Society meeting reported that a system designed to ensure at least one contact results in four or five actual contacts for the average molecule, and thereby reduces the probability of a molecule passing through to truly negligible proportions.

Although heated grids for oil decomposition have been discussed in literature some time ago, their effect has not been evaluated. To be efficient, they should be tight, in which case they will occupy a large area of the inlet duct and consequently decrease the pumping speed. There is a possibility that such heaters may drive the products of oil decomposition into the chamber. It would be perhaps better to place such baffles below the liquid nitrogen trap rather than above as used by the authors. In addition, the heaters in Fig. 14d, e, and h appear to heat the optical baffles above them by radiation and therefore make those baffles even less effective. The copper foil traps shown in Fig. 14c are only effective after a thorough bakeout and they have been proven only in small glass systems. It is not surprising therefore that they were not helpful as used by the authors.

In addition, it would be desirable for friction experiments under space conditions to have a higher vacuum than produced in the system described in the paper. Even with a liquid helium condensing coil, the authors achieved only 2×10^{-7} mm Hg, which is about three orders of magnitude higher than the capabilities of presently available ultrahigh vacuum systems with oil diffusion pumps.

Following are some references which indicate that vacuum essentially free of heavier hydrocarbons can be obtained with oil diffusion pumps.

1. "The Measurement of Low Partial Gas Pressure with the Mass Spectrometer" by H. W. Drawin and C. Brune (Atlas-Werke AG, Germany) from *Vakuum-Technik*, No. 3 (1960). The following partial pressures were obtained with mercury diffusion pumps:

Hydrogen, 1×10^{-9} mm Hg; Water, 1×10^{-10} mm Hg; Carbon monoxide, 5×10^{-10} mm Hg; Nitrogen, 5×10^{-10} mm Hg; Carbon dioxide, less than 1.5×10^{-9} mm Hg; Hydrocarbons, less than 1×10^{-10} mm Hg; Mercury, less than 1×10^{-10} mm Hg; Total pressure, less than 3.3×10^{-9} mm Hg. Using oil diffusion pumps, very similar results were obtained at total pressure of 3×10^{-9} mm Hg. The authors say: "... With both pump types, about the same ultimate partial pressures can be obtained with the exception of hydrogen. The partial pressure of hydrogen was at least ten times higher with the oil diffusion pumps compared to the mercury pumps." It may be noted also that reduction of foreline pressure of a diffusion pump reduces the amount of hydrogen on the high vacuum side.

2. N. Milleron (Lawrence Radiation Laboratory, University of California, Livermore, Calif.) at the *First Symposium on Surface Effects on Spacecraft Materials, Palo Alto, California* (May, 1959). "Diffusion Pumps have been demonstrated to be inherently clean . . . First, diffusion pumps, both mercury and oil, can be completely trapped. No oil or cracked hydrocarbon spec-

trum, and no mercury, down to partial pressures of 10^{-11} mm Hg for oil and 10^{-16} mm Hg for mercury is detected."

3. "Mass Spectrometer Investigations in the Ultrahigh Vacuum" by P. Jahn and J. Zahringer (Max-Planck Institute for Nuclear Physics, Heidelberg), *Vakuum-Technik*, No. 10, 5, p. 138 (June, 1961).

"A very sensitive mass spectrometer—allowing the measurement of partial pressures below 10^{-12} torr—was used to investigate the composition of the residual gas in a vacuum system, as obtained with oil diffusion pumps plus nonrefrigerated baffles, and with an ion-getter pump. The spectrum of the residual gas was checked daily during several weeks whereby different types of pumps, baffles and pumping fluids have been used."

"The investigations have shown that the combination two-stage pump plus silicone-ultra fluid plus aluminum oxide trap has been proven suitable for production of ultrahigh vacuum. The pressure remains constant for many weeks at its initial value and the residual gases from the pump are only methane and hydrogen."

In general, experiments which greatly depend on surface conditions must be conducted with very careful vacuum design and techniques, or contamination is possible with both diffusion pumps and ion-gettering pumps when used together with mechanical pumps.

AUTHORS' CLOSURE:

The comments by Messrs. M. H. Hablanian and E. G. Jackson on vacuum technology with diffusion pumps are most welcome even though they do not pertain to the body of the paper. The appendix to this paper to which they refer was included primarily so that other investigators would not have to duplicate this effort. It is reasonable to expect that the problem of back migration *can* be solved. The efforts described in the appendix, however, did *not* solve the problem. Rather than to spend more effort, the problem was avoided by using an ion pump. The ion pumped system is not free from problems but fortunately they are different than the one of primary concern here.

The references cited by the discussers are indicated to suggest that systems "*essentially*" free of back migration can be obtained. The problem in friction, wear, and lubrication studies is that systems must be *absolutely* free of organic contamination. In visits to a number of laboratories no diffusion pumped systems were observed that could be considered absolutely clean. No doubt those systems included many of the design considerations advanced by the discussers. One laboratory indicated that a diffusion pumped system with a zeolite trap was completely free of back migration. The effectiveness of such a trap has not been verified by the authors, however, it merits consideration where only diffusion pumped systems are available.

One specific point raised by the discussers concerned the high pressure achieved with liquid helium cryopumping. That pressure (2×10^{-7} mm Hg) was obtained by design rather than as a minimum pressure. The point made was that pressure measurements alone are not an adequate means of defining space simulation. It is important to define molecular and atomic species present in the chamber and to measure partial pressures. Using liquid helium to the limit of its cryopumping capabilities in the apparatus, we achieved pressures in the 10^{-9} mm Hg range. The pressures were measured with a nude hot cathode ionization gage adjacent to the experimental specimens. Using other gages of types more commonly employed by others including the discussers, lower values would be obtained.

for ASLE-ASME meeting,
Chicago, Ill., October 1961

FRICTION, WEAR, AND EVAPORATION RATES OF VARIOUS

MATERIALS IN VACUUM TO 10^{-7} mm Hg

By Donald H. Buckley,¹ Max Swikert,² and Robert L. Johnson³

ABSTRACT

Evaporation data on soft metals, lubricating inorganic compounds, and various reference materials are reported for temperatures from 75° to 1000° F in vacuum as low as 10^{-7} mm Hg. Observations on modes of vacuum degradation (e.g., evaporation or dissociation) and methods of experimentation are related. Friction and wear data are presented for several unlubricated metals (e.g., type 440-C steel) and metals coated with inorganic (e.g., MoS_2 , CaF_2) as well as with soft metal films in vacuum at ambient pressures between 10^{-6} and 10^{-7} mm Hg.

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The requirements for bearings and seals to operate in the environment of space dictate a new area for lubrication research. The low ambient pressures encountered in space can be expected to influence the behavior of oil, grease, and solid-film lubricants. The property of these materials most significantly affected by low ambient pressures is the evaporation rate. Various investigators have therefore measured the evaporation rates of oils and greases in vacuum as one method of establishing their relative merit for space applications (1-3). The results of this work have given some indication as to the oils and greases with the greatest stability at reduced

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1163
(5)

ambient pressures. Only limited experimental work, however, has been reported in the literature for inorganic solids and soft metals which have potential use as solid lubricant films or coatings for hard alloy substrates (e.g., (4)). In general, the evaporation rates of these materials would be lower than those of oils and greases. These films might therefore be very attractive as lubricants for high vacuum service.

The lack of oxygen in outer space creates another problem for lubrication systems, namely, the absence of the protective metal surface oxides normally encountered in air. In the absence of these or other protective surface films, mass metal transfer, welding, and high coefficients of friction may be experienced for metals in sliding contact (5-7). Some data are presented in the literature for the friction and wear of metals and soft alloys in vacuum (e.g., (8)). In general, these data were obtained with conventional oil diffusion pumps in the vacuum systems. One of the problems associated with such systems is that of back-migration of oil vapors from the pump to the test chamber. This back-migration even with cold traps and baffles can serve as a source of specimen contamination and result in a pronounced reduction in friction and wear.

There is a need for data on the evaporation rates of lubricants and also on the friction and wear characteristics of potential bearing and seal materials; these data must be obtained in a vacuum environment to facilitate the proper selection of lubricants and slider materials intended for use in space. The friction and wear data should be obtained in systems which are free from sources of specimen contamination like that associated with the oil diffusion pumps.

The objectives of this investigation were to determine in vacuum (10^{-6} to 10^{-7} mm Hg): (1) the evaporation rates for various organic and inorganic lubricants, (2) the evaporation rates of solid lubricant coatings, (3) the friction and wear properties of unlubricated slider materials for reference, and (4) the friction and wear properties of these slider materials coated with solid lubricant films. Evaporation-rate experiments were used to select the most promising solid lubricants for use as surface films on slider materials in vacuum. Evaporation rates were measured at temperatures from 55° to 1000° F. Friction and wear experiments were conducted with a 3/16-in.-radius rider sliding on a $2\frac{1}{2}$ -in.-diameter disk specimen at surface speeds of 390 ft/min. The rider was loaded against the disk with a 1000-g load. The duration of the experiments was 1 hr.

MATERIALS

The specimens used in the evaporation experiments of this investigation were oils, greases, inorganic solids, metals, and solid lubricant coatings. The oils included MIL-L-7808 (di-2-ethyl hexyl sebacate), two mineral oils (viscosities of 380 and 235 centistokes at 100° F) designed for high-temperature use, and a polyphenyl ether (1-(p- α -cumyl phenoxy)-4-phenoxy benzene). The ether was a solid at room temperature. The greases included were a radiation-resistant grease and two mineral-oil-base greases with phthalocyanine thickeners. The inorganics included chemically pure materials obtained from commercial supply houses: cadmium iodide (CdI_2), cobalt chloride (CoCl_2), nickel fluoride (NiF_2), lead oxide (PbO), calcium fluoride (CaF_2), barium fluoride (BaF_2), tungsten disulfide (WS_2), and molybdenum disulfide (MoS_2). The nickel bromide (NiBr_2) could not be readily obtained commercially and was therefore prepared in the laboratory. The metals

included were cadmium (Cd), indium (In), zinc (Zn), magnesium (Mg), tin (Sn), gallium (Ga), lead (Pb), and silver (Ag). Two organics other than the oils and greases for which evaporation rates were measured were polytetrafluoroethylene (PTFE) and epoxy (diglycidyl ether of bisphenol A with a diethylene triamine catalyst).

The alloys used in the friction and wear experiment with their compositions are presented in the following table:

Alloy Compositions

Metal	Composition												Hardness, Rockwell
	Fe	Ni	Co	Cr	C	Mo	W	Ti	Si	Mn	Al	Other	
52100	Bal. of comp.			1.3 to 1.6	0.95 to 1.10				0.20 to .35	0.25 to .45		P, 0.025 S, 0.025	R _C 60-63
440-C	Bal. of comp.			16.0 to 18.0	0.95 to 1.20	0.75			1.0 Max.	1.0		P, S	R _C 54
Ni-Cr	0.250	Bal. of comp.	11.0 to 12.0	18.0 to 20.0	0.06 to .12	9.0 to 10.5		3.2	0.010	0.050	1.50		R _C 43
Ni-Cr-Fe	5.0 to 9.0	70.0		15.0	0.08				0.50			S, Cb Al, Mn	R _C 29
Cobalt alloy	3.0	2.5	43.0	31.0	2.4		17.0		1.0				R _C 52-63
Ni-bonded TiC and CbC*		25.0			13.2	5.0		52.0				Cb, 4.5; Ta, 0.3	R _A 89

*Ti and Cb are present as mixed carbides.

The molybdenum disulfide coatings used were coatings of the following types: silicon resin bonded, epoxy-phenolic resin bonded, ceramic bonded, and a metal matrix coating. The coatings were applied to 0.003-in.-thick

metal tabs for evaporation experiments. The thickness of the applied coating was .0.001 in. In the friction and wear experiments, however, the coating thickness on the disk specimens was 0.0002 to 0.0003 in.

APPARATUS

The apparatus used in this investigation is presented in Figs. 1(a) to (c). The evaporation apparatus consisted of a commercial bell-jar vacuum system with a 4-in. oil diffusion pump system. Pressures in the 18-in.-diameter chamber were measured with thermocouple and hot cathode ionization gages. Inside the bell-jar chamber just above the throat of the diffusion pump, a large plate was used to support a heater and the test specimen. The heater consisted of a housing of machinable ceramic and a wire-wound tungsten heating element. The specimen was placed on the heating element. A thermocouple was positioned on the top edge of the specimen for temperature measurement and control.

Approximately 4 in. above the specimen was a condensing shield. This shield was cooled with a liquid-nitrogen coil, and the temperature measured at the center of the condensing shield when liquid passed through the coil was -204° F. On the side of the shield facing the specimen was a copper tab for making X-ray analyses of condensate composition.

The bell-jar evaporation apparatus was modified for use with a continuous recording balance. The apparatus with associated modifications is presented in Fig. 1(b). The weighing mechanism of a commercially available electronic balance was mounted on a support in the upper part of the bell jar. Suspended from the balance beam was a thin wire that extended from the balance beam through the condensing shield to a stirrup that supported the specimen. The specimen and stirrup were suspended in a cylindrical furnace. The walls of

the furnace contained the heating elements of wound tungsten wire. A dummy specimen with thermocouple placed beneath the test specimen was used to measure and control specimen temperatures. The dummy specimen technique has been employed by others in similar experiments (9). Weight changes in the specimen were continuously recorded on a strip chart.

The vacuum friction and wear apparatus is shown schematically in Fig. 1(c). The basic elements of the apparatus were a $2\frac{1}{2}$ -in.-diameter disk and a $3/16$ -in.-radius rider specimen. The disk was rotated by means of an induction motor with a canned rotor and stator. The enclosure of rotor and stator permitted a vacuum in the motor chamber. A vacuum valve connected the motor chamber with a mechanical pump system. The system included a liquid-nitrogen cold trap to inhibit oil migration from the pump. The specimen chamber containing the disk and rider was attached to the motor. The motor drive shaft extended into the specimen chamber with the disk specimen bolted directly to the end. The only seal between chambers was that afforded by a ball bearing mounted on the drive shaft. Since the presence of the ball bearing did not provide a vacuum seal, the ionization pump pumped both chambers. When the pressure in the specimen chamber was 10^{-7} mm Hg, it was approximately 10^{-4} mm Hg in the motor chamber.

The rider specimen was supported in the specimen chamber by a retaining arm that was gimbal and bellows mounted to the chamber. A linkage at the end of the retaining arm away from the rider specimen was connected to a strain-gage assembly. The assembly was used to measure frictional force. Load was applied through a dead-weight loading system.

Attached to the lower end of the specimen chamber was a vacuum valve. The side of the valve away from the chamber had a T-section. On the lower

portion of the T was connected a 400-1/sec ionization pump. The remaining portion of the T connected with a vacuum valve and a mechanical pump using liquid-nitrogen cold traps.

The first high-vacuum pump to be used with the vacuum friction and wear apparatus was a conventional oil diffusion pump. In initial experiments it was established, however, that specimen contamination by oil vapors had occurred (see Appendix A). Various liquid-nitrogen cold-trap designs were employed to eliminate this problem. Subsequent specimen examinations, however, indicated surface contamination. Combinations of cold traps, optical baffles, and heating grids for decomposition of oil vapors were then tried with little or no success. Since, even with the best trapping techniques, the possibility of specimen contamination did exist, the diffusion pump was replaced by an ionization pump. This pump employs no fluids or vapors in its operation.

EXPERIMENTAL PROCEDURE

The procedure used in the evaporation studies consisted of first preparing the experimental specimens. This was accomplished for the solids by oven-drying and dehydrating the materials. When the powders were dry, small charges were placed in a mold and compressed into tablet form under a 10,000-psi compression force. When the specimens were removed, they measured 5/8 in. in diameter and were approximately 1/8 in. thick. The tablets were then placed into a small preweighed glazed ceramic dish that had side walls of 1/8 in. and inside diameter just large enough to accommodate the tablet specimen. The specimens and holders were then stored in a desiccator until ready for use. The oils and greases were placed directly in the dishes with the specimen filling the dish.

Prior to an experiment the specimens were removed from the desiccator and weighed. The specimen and holder were then placed into the vacuum chamber, and the chamber was evacuated. At a pressure between 10^{-6} and 10^{-7} mm Hg the experiment was started. In those experiments conducted above room temperature, the specimens were heated by a tungsten heater on which the specimen was placed.

The evaporation-rate experiments made with solid lubricant coatings were run using the electronic balance with a continuous measure of weight change with time. During an experiment a tab was placed on the stirrup that was suspended from the balance at the top end of the furnace, and the chamber was then evacuated. At a pressure between 10^{-6} and 10^{-7} mm Hg the evaporation experiments were started.

The disk and rider specimens used in friction and wear experiments were finish-ground at 4 to 8 microinches. Before each experiment the disk and rider were given the same preparatory treatment. This treatment consisted of (1) a thorough rinsing with acetone to remove oil and grease, (2) polishing with moist levigated alumina on a soft cloth, and (3) a thorough rinsing with tap water followed by distilled water. For each experiment (data point) a new set of specimens was used. In those experiments in which disks with coatings were used, the coatings were applied after the cleaning procedure. Plated specimens were solvent-cleaned, and bonded coatings were carefully handled following coating in order to avoid contamination.

The specimens were then placed in the apparatus. Mechanical pumps with liquid-nitrogen cold traps were used to obtain a pressure of 10^{-4} mm Hg in the specimen chamber. In the startup of the ionization pump, ionization of chamber gases was produced by the pump. This ionization generally persisted

for 20 to 30 min. In the event that oil molecules had migrated through the liquid-nitrogen cold traps of the mechanical pumps prior to being valved off from the specimen chamber, the ionization would decompose them.

When the specimen chamber pressure was between 5.0×10^{-7} and 2.0×10^{-6} mm Hg as measured by a hot cathode ionization gage and by the ionization pump current, the friction experiment was started. A 1000-g load was applied to the rider loading it against the disk. Friction force was continuously measured and recorded on a strip chart. The wear was measured as volume loss of the rider specimen upon completion of 1 hr of running. The wear to the disk specimen surface was recorded with a surface profile measuring device.

RESULTS AND DISCUSSION

Evaporation Data

The evaporation rates for some low-vapor-pressure oils and greases were determined in vacuum at various temperatures. The results obtained in these evaporation experiments are presented in Fig. 2. The sebacate MIL-L-7808 bubbled continuously while evaporating. The high-temperature mineral oils (viscosities of 380 and 235 centistokes at 100° F) did not exhibit this characteristic, and the surfaces were rather quiescent during evaporation at 55° and 200° F. The ether (1- α -cumyl phenoxy-4-phenoxy-benzene was a solid at 55° F and exhibited no detectable weight change in 60.0 hr. At 200° F the ether was in a liquid state, and the evaporation rate was quite high (2.5×10^{-6} g/cm²/sec).

The greases appeared to lose the base oil with increase in temperature, leaving the bulk of the thickener behind. The evaporation experiments were terminated before complete evaporation of the base oil had occurred. This was found desirable in order to avoid a change in evaporation rates once the

base stock had completely evaporated and the thickener with its own characteristic rate began to evaporate. The radiation-resistant grease exhibited a very low rate of evaporation at 55° F. At 200° F, however, the rate increased and the specimen became rubbery. The greases using phthalocyanine as a thickener both showed evidence of the thickener evaporating with the base stock, as determined by examination of the condensate on the liquid-nitrogen condensing shield. The evaporation rates of the greases were tolerable at 200° F; however, at 350° F the rate was quite high.

The evaporation rates for compressed disks of various lubricant coating constituents were determined, and the results obtained in vacuum at various temperatures are presented in Fig. 3. The nickel fluoride showed no evidence of evaporation at 55° or 200° F. At 350° F, however, the nickel fluoride dissociated to metallic nickel and fluorine. The specimen surface changed from yellow-green to a black color. X-ray analysis of the surface indicated the black film to be metallic nickel. The lead oxide also dissociated at 350° F to metallic lead and oxygen. X-ray analysis indicated the presence of lead on the specimen surface. The cobalt chloride sample exhibited a color change from blue to green at 500° F; however, the evaporation rate was low. X-ray analysis of the sample indicated its composition to be cobalt chloride (CoCl_2). The cobalt chloride, cadmium iodide, and nickel bromide evaporated and condensed on the condensing shield as cobalt chloride, cadmium iodide, and nickel bromide, respectively. The epoxy composition (diglycidyl ether of bisphenol A with a catalyst of diethylene triamine) showed evidence of decomposition at 350° F.

The evaporation rates for molybdenum disulfide, tungsten disulfide, calcium fluoride, and barium fluoride at temperatures to 1000° F are presented in Fig. 4. The weight losses for these materials in vacuum at elevated temperatures were very low, and in general these materials appeared to be the most stable of the inorganics tested. The rates for molybdenum disulfide, calcium fluoride, and barium fluoride were extremely close, and a single curve was drawn to represent the evaporation rate of these three materials. The molybdenum and tungsten disulfide had to be heated in vacuum prior to an experiment to remove adsorbed gases.

The evaporation rates for polytetrafluorethylene (pure polymer without plasticizer) were determined at various ambient temperatures and pressures. The results obtained in these experiments are presented in Fig. 5. While the polytetrafluorethylene exhibited some weight change at various ambient pressures, this weight change was extremely small. The evaporation rate of polytetrafluorethylene at various temperatures was determined at an ambient pressure of 8.0×10^{-7} to 2.0×10^{-6} mm Hg. The evaporation rate of polytetrafluorethylene was low to 350° F; at 650° F the rate was high. Polytetrafluorethylene begins to decompose at temperatures above 500° F; this may well account for the high evaporation obtained at 650° F.

The evaporation rates for various metals were determined in vacuum over a range of temperatures, and the results obtained in these experiments are presented in Figs. 6 and 7. Cadmium and zinc exhibited relatively high rates of evaporation. The rates were higher than obtained with polytetrafluorethylene. Lead, tin, and silver had the lowest rates of evaporation of the soft metals investigated.

When the evaporation rates determined experimentally in this investigation are compared with data calculated from vapor pressure presented in the literature (10), the results appear to be in good agreement (Fig. 6). Any differences observed could be due not only to experimental error but to error in the calculated values. The calculated values are based on thermodynamic relations where an error of 10 percent is not unlikely. Since the evaporation rate can be determined from vapor pressure, the reverse must also be possible. The use of evaporation rates to determine vapor pressure can be accomplished for pure metals by a simple equation:

$$P_{\text{mm}} = 17.14 G \sqrt{\frac{T}{m}}$$

where

P_{mm} = vapor pressure in mm Hg

G = evaporation rate in $\text{g}/\text{cm}^2/\text{sec}$

T = temperature in $^{\circ}\text{K}$

m = molecular weight in grams

The vapor pressure of zinc is determined from its evaporation rate at 500°F :

$$P_{\text{mm}} = 17.14(2.0 \times 10^{-6}) \sqrt{\frac{533}{65.38}}$$

$$P_{\text{mm}} = 9.81 \times 10^{-5}$$

The vapor pressure of zinc at 500°F presented in the literature (10) is 1.0×10^{-4} mm Hg.

Evaporation-rate experiments were conducted with four molybdenum disulfide coatings in vacuum at various temperatures. An electronic balance was used to continuously measure weight changes with time. The results obtained in these experiments are presented in Fig. 8. In general, there appears to

be a break in evaporation curves for all coatings; this result indicates a change in evaporation rate. This change may be due to two different evaporation rates, one for the binder, and the other for molybdenum disulfide itself.

Friction and Wear Data

Unlubricated metals. - Friction and wear experiments were conducted in air and vacuum with five alloy combinations. The results obtained in these experiments are presented in Fig. 9. The friction and wear values for 52100 sliding on itself were lower in vacuum than in air. Examination of the disk surfaces after the experiments indicated a change in the wear mechanism (Figs. 10(a) and (b)). Free metal wear particles are oxidized in air, and the resulting metal oxides on the particles prevent adherence to the base metal. At an ambient pressure of 10^{-6} mm Hg, however, where oxygen availability was appreciably reduced, the wear surface of the disk showed evidence of considerable metal transfer as nascent wear particles readily adhered to the parent metal. The surface profile tracings of Fig. 10(b) indicate "buildup" on the disk surface. This was apparently due to the transfer of metal from the rider to the disk specimen.

The low friction coefficients experienced for 52100 steel in vacuum (10^{-6} mm Hg) may be accounted for in that, even at 10^{-6} mm Hg, oxygen is present. Bowden and Tabor (11) have indicated that copper will oxidize as rapidly at 10^{-3} mm Hg pressure as it will at atmospheric pressure; also, it has been established elsewhere that, at 10^{-6} mm Hg, a clean metal surface will adsorb a monolayer of gas in a second. Since some oxygen is available at 10^{-6} mm Hg, it is probable that as pieces of metal, which are quite hot, transfer from one specimen surface to the other, localized oxidation of the transferred metal occurs. With the limited oxygen available the lower oxides

of iron, FeO and Fe_3O_4 , would form on ferrous metal and result in a friction coefficient lower than normally encountered in air at atmospheric pressure (12) where Fe_2O_3 is one of the oxides present. Results obtained at pressures lower than 10^{-6} mm Hg should differ from those obtained at 10^{-6} mm Hg.

The coefficient of friction for 52100 tool steel sliding on 52100 tool steel at various ambient pressures is presented in Fig. 11. The friction decreased from 0.45 at 760 mm Hg pressure to 0.2 between 10^{-1} and 10^{-2} mm Hg. The friction began to increase again at 10^{-4} mm Hg, and at 5.0×10^{-7} mm Hg it was 0.375.

The friction and wear results obtained with 440-C stainless steel sliding on itself in vacuum were about the same as in air (Fig. 9). Examination of surface photomicrographs and profile tracings of the disk wear area (Figs. 10(c) and (d)) indicate that wear debris was transferred to the disk specimen.

The friction and wear of a cobalt-base alloy were determined in vacuum and air, and the results obtained are presented in Fig. 9. The friction and wear were lower in vacuum than in air, and examination of the surface topography indicated no evidence of mass metal transfer in vacuum (Fig. 10(f)). Similar results have been observed for this particular alloy in inert and reducing atmospheres (data in process of publication). This particular alloy is an air cast material and has about 1.0 percent silicon in its composition. The influence of silicon on the friction and wear properties of materials is discussed in (13). It was established in (11) that small quantities of silicon rather markedly reduced the friction and wear normally encountered for alloys in inert and reducing atmospheres. This same mechanism could be expected to apply in vacuum.

The friction and wear properties of a vacuum-melted nickel-base alloy (Ni-Cr) were next determined. The results obtained in air and vacuum are presented in Fig. 9. The coefficient of friction in vacuum was greater than in air. The rider wear, however, was about the same in both environments. Examination of this wear area on the disk specimen indicated metal transfer in vacuum (Fig. 10(h)). From the photomicrograph it appears that pieces of metal were transferred from rider to disk and then smeared in the direction of sliding. Though the rider wear was nearly the same in air and in vacuum, the wear mechanism on the disk surfaces was quite different, as indicated by comparison of the photomicrograph and surface profile traces of Figs. 10(g) and (h).

The friction and wear values for a nickel-bonded titanium and columbium carbide cermet sliding on itself is presented in Fig. 9. For this particular material both friction and wear were higher in vacuum than in air. The photomicrograph and surface profile tracing of Fig. 10(j) indicate evidence of metal transfer to the disk surface in vacuum.

Lubricated metals. - The mechanism of wear for metals in vacuum indicates that stable films should be employed to lubricate these materials. Since molybdenum disulfide has been shown to be a good dry film lubricant in air and has very low evaporation rates in vacuum, molybdenum disulfide coatings using various binders were used as solid-film lubricants for 440-C stainless steel in vacuum. The friction and wear results obtained with 440-C sliding on 440-C coated with various molybdenum disulfide films in vacuum are presented in Fig. 12. From the results obtained, it appears the lubricant binder plays an important role in the friction and wear results obtained with molybdenum

disulfide coatings. The best friction and wear were obtained with an epoxy-phenolic and a silicone resin-bonded molybdenum disulfide coating. The metal matrix was less satisfactory, and the ceramic bonded gave the poorest results with a continuous friction coefficient of about 0.3 over an entire 1 hr of operating. These results, however, were obtained at room temperature, and the epoxy-phenolic coating may not appear as promising at elevated temperatures.

Friction and wear experiments were made with other solid film, and the results obtained are presented in Fig. 13. A lead oxide - silicon dioxide coating developed for high-temperature use in air was applied to 440-C stainless steel and was run in vacuum. A friction coefficient of about 0.17 was obtained at 10^{-6} to 10^{-7} mm Hg ambient pressure. In air at room temperature, with a sliding velocity of 430 ft/min, this particular coating had a friction coefficient of 0.3 (14). In vacuum the surface became sufficiently heated without an external heat addition to give a friction coefficient similar to that obtained in air at 250° F in (14).

A calcium fluoride solid lubricant coating developed for high-temperature applications was applied to a Ni-Cr disk. A Ni-Cr-Fe rider was used against the disk in vacuum. The friction and wear obtained are presented in Fig. 13. The coating gave a friction coefficient of 0.18 over a 1-hr period, and the wear to the rider specimen was low. The friction coefficient obtained with this coating in vacuum was lower than obtained in air at the same temperature.

In the literature, instances are cited where thin, soft metal films were used as protective coatings for bearings run in vacuum (4), (15). Various soft metals were therefore applied to 440-C stainless steel substrates to determine their influence on the friction and wear of these substrates in

vacuum. The results obtained with a 440-C stainless steel rider sliding on the thin metal film (0.0004 in. thickness) coated 440-C disks are presented in Fig. 13. The friction coefficient for lead, gold, and silver was less than 0.1 while tin was about 0.14. The wear for 440-C stainless steel was low with all four coatings. The thin metal films appear to have properties when applied to hard substrates that make them potential candidates as solid-film lubricants for use in vacuum.

SUMMARY OF RESULTS

Evaporation rates, and friction and wear were obtained at a pressure of 10^{-6} to 10^{-7} mm Hg. Since the pressures encountered in space may be many orders of magnitude lower (approx. 10^{-14} mm Hg), the results obtained in similar experiments at lower pressures may differ somewhat from those obtained herein. The results presented here, however, do give some indication of the effects of reduced ambient pressures on lubricants, friction, and wear. The results obtained in this study are summarized as follows:

1. The use of MoS_2 coatings (epoxy-phenolic resin and silicone resin bonded 0.0002 to 0.0003 in. thickness) provided effective solid lubricant films for 440-C stainless steel at pressures of 10^{-6} to 10^{-7} mm Hg. Both friction and wear were extremely low with these coatings.

2. Some metals (Ag, Sn, Au, and Pb) when applied to substrates such as 440-C stainless steel (0.0004 in. thickness) appreciably reduce the friction and wear normally encountered with this alloy in vacuum (10^{-6} to 10^{-7} mm Hg).

3. The friction and wear results obtained with various metals in vacuum (10^{-6} to 10^{-7} mm Hg) indicate a wear mechanism unlike that encountered in air. In general the wear in vacuum was characterized by mass metal transfer. The friction coefficients were lower than obtained in air for some alloys

(e.g., 52100) and considerably higher for others (e.g., Ni-Cr alloy and Ni-bonded TiC and CbC). Operation at lower pressures might be expected to further alter the wear mechanism based on oxygen availability.

4. The evaporation data for some inorganics (CaF_2 and MoS_2) and metals (Ag and Sn) indicate they have potential for use as lubricants in vacuum (10^{-6} mm Hg) to temperatures as high as 1000°F .

5. The evaporation data indicate that some oils and greases may be used as lubricants at pressures of 10^{-6} mm Hg for short periods of time provided the ambient temperature is low (55°F).

APPENDIX A

A STUDY OF THE EFFECTIVENESS OF VARIOUS TRAPPING DEVICES IN STOPPING BACK-MIGRATION OF DIFFUSION PUMP OILS

The vacuum friction and wear apparatus used in this investigation originally employed an oil diffusion pump to obtain high vacuum. The oil diffusion pump has associated with it the undesirable characteristic of permitting oil vapors to migrate from the pump. In the vacuum literature it has been suggested that this problem can be eliminated by the use of baffles and/or cold traps between the oil diffusion pump and experimental chambers. Numerous cold-trap and baffle designs have been reported in the literature and users of oil diffusion pumps have employed these and other modifications in vacuum systems. There appears, in general, to be very little if any standardization on cold-trap and baffle designs. Various cold traps and baffles were therefore tried in the vacuum friction and wear apparatus in an attempt to eliminate back-migration of oil vapors.

The first step taken was to place a liquid-nitrogen spiral cold trap in the apparatus between the experimental chamber and the oil diffusion pump. A

schematic of this cold trap is shown in Fig. A1(a). The gas being pumped had to travel a spiral path along liquid-nitrogen-cooled walls. A friction experiment was conducted with a disk and rider specimen in the experimental chamber. The friction coefficient obtained with clean 440-C stainless steel was in a region (0.1) associated with effective boundary lubrication. The disk surface was checked with distilled water after the experiment; observations of high contact angle and lack of wettability gave evidence of an oil film.

An optical baffle plate was then added between the cold trap and specimens in an attempt to eliminate the contamination. The baffle design used is shown in Fig. A1(b). Friction experiments with 440-C stainless steel again gave a friction coefficient indicative of effective boundary lubrication. Examination of the disk surface with distilled water showed evidence of a contaminated surface.

The use of a honeycomb copper structure as a baffle has been reported in the literature as a means of trapping oil molecules. A baffle of this design was fitted to the optical baffle, and the unit was placed in the specimen chamber above the liquid-nitrogen cold trap (Fig. A1(c)). This arrangement was not effective in preventing specimen contamination by oil vapors.

Another approach to eliminating the back-migration problem considered was that of using heat to decompose oil vapors migrating to the specimen chamber. To achieve this, a heater was attached to the optical baffle previously used. The optical baffle with heater placed between it and the liquid-nitrogen cold trap is shown in Fig. A1(d). This arrangement was also inadequate in eliminating specimen contamination, as ascertained by friction experiments with clean 440-C stainless steel and wettability checks. The heater plate

was then replaced by a heater grid of tungsten coils. These coils were heated to an orange-yellow heat in an attempt to decompose oil vapors entering the specimen chamber. The heater grid and optical baffle used are shown in Fig. Al(e). This arrangement was also unsatisfactory.

A tungsten-wound heater coil was then placed in the specimen chamber directly beneath the disk and rider in an attempt to keep vapors from the specimen surfaces (Fig. Al(f)). This arrangement was insufficient, and the specimens again exhibited evidence of surface contamination.

The design of the optical baffle previously used was altered, and the new optical baffle is shown in Fig. Al(g), where it was used in conjunction with the liquid-nitrogen cold trap. In Fig. Al(h) a tungsten heater grid was added below the baffle. Neither of these systems accomplished the elimination of oil contamination as determined by friction coefficients and specimen surface examination.

The next approach to the back-migration problem considered was to remove the expansion bellows of the apparatus located between the specimen chamber and the liquid-nitrogen cold trap and to replace it with a heater-baffle assembly. This assembly can be seen in Fig. Al(i). It consisted of two water-cooled optical baffle plates with a tungsten-wound heater grid between the plates. The temperature of the space between the tungsten grids maintained during diffusion pump operation was 1000° F. The outside walls of the chamber were water-cooled. This assembly in conjunction with the liquid-nitrogen cold trap minimized the specimen contamination. Examination of the disk surface with distilled water indicated a contact angle less than previously obtained; however, the water still would not wet the metal surface, and the coefficient of friction for 440-C stainless steel was about 0.2. An addition was then

made to the system, namely, the optical baffle used in earlier trapping designs. The complete trapping system can be seen in Fig. A1(j). The results obtained were about the same as with the arrangement of Fig. A1(i).

A liquid-nitrogen-cooled plate type chevron baffle was also considered for these experiments. This configuration was not, however, used because experimental evidence by others at Lewis Research Center showed that it also allowed test-chamber contamination.

The experiences related here were obtained with a common diffusion pump oil composed of petroleum hydrocarbons. Separate experiments were conducted with additional diffusion pump oils including a silicone and a sebacate. In no case was back-migration of diffusion pump oil completely eliminated.

The inability to completely eliminate the back-migration of oil vapors from the oil diffusion pump resulted in the replacement of the oil diffusion pump with an ionization pump. This pump employs no fluid or vapors in its operation.

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FIGURE LEGENDS

Fig. 1(a). Vacuum apparatus for bulk evaporation studies.

Fig. 1(b). Vacuum evaporation apparatus with balance for continuous rate measurements.

Fig. 1(c). Vacuum friction and wear apparatus.

Fig. 2. Evaporation rates for various oils and greases in vacuum. Ambient pressure, 8.0×10^{-7} to 2.0×10^{-6} mm Hg.

Fig. 3. Evaporation rates for possible coating constituents in vacuum. Ambient pressure, 8.0×10^{-7} to 2.0×10^{-6} mm Hg.

Fig. 4. Evaporation rates for various inorganic compounds in vacuum. Ambient pressure, 8.0×10^{-7} to 2.0×10^{-6} mm Hg.

Fig. 5. Evaporation rate of PTFE at various temperatures and pressures.

Fig. 6. Evaporation rates for various metals in vacuum. Ambient pressure, 8.0×10^{-7} to 2.0×10^{-6} mm Hg.

Fig. 7. Evaporation rates for various metals in vacuum. Ambient pressure, 8.0×10^{-7} to 2.0×10^{-6} mm Hg.

Fig. 8. Evaporation rate of various MoS₂ coatings in vacuum. Ambient pressure, 1.0 - 2.0×10^{-6} mm Hg; 0.001-in. MoS₂ coating on Ni-Cr alloy.

Fig. 9. Friction and wear of various alloys in air and in vacuum. Vacuum, 5.0×10^{-7} to 2.0×10^{-6} mm Hg; sliding velocity, 390 ft/min; load, 1000 g; duration of run, 1 hr.

(a) Disk specimen, 52100 tool steel in air.

(b) Disk specimen, 52100 tool steel in vacuum.

(c) Disk specimen, 440-C stainless steel in air.

(d) Disk specimen, 440-C stainless steel in vacuum.

Fig. 10. Photomicrographs and surface profile tracings of wear areas on disk specimens, load, 1000 g; sliding velocity, 390 ft/min; duration of run, 1 hr; X20.

(e) Disk specimen, cobalt-base alloy in air.

(f) Disk specimen, cobalt-base alloy in vacuum.

(g) Disk specimen, Ni-Cr-Al alloy in air.

(h) Disk specimen, Ni-Cr-Al alloy in vacuum.

Fig. 10. Continued. Photomicrographs and surface profile tracings of wear areas on disk specimens, load, g; sliding velocity, 390 ft/min; duration of run, 1 hr; X20.

(i) Specimen and rider, Ni-bonded TiC and CbC cermet in air.

(j) Specimen and rider, Ni-bonded TiC and CbC cermet in vacuum.

Fig. 10. Concluded. Photomicrographs and surface profile tracings of wear areas on disk specimens, load, g; sliding velocity, 390 ft/min; duration of run, 1 hr; X20.

Fig. 11. Coefficient of friction for 52100 sliding on 52100 at various ambient pressures. Sliding velocity, 390 ft/min; load, 1000 g; temperature, 75° F.

Fig. 12. Friction and wear of 440-C on 440-C with various MoS₂ films. Ambient pressure, 8.0×10^{-7} to 2.0×10^{-6} mm Hg; sliding velocity, 390 ft/min; load, 1000 g; duration of run, 1 hr.

Fig. 13. Friction and wear of alloys with various coatings in vacuum. Ambient pressure, 8.0×10^{-7} to 2.0×10^{-6} mm Hg; sliding velocity, 390 ft/min; load, 1000 g; duration of run, 1 hr.

Fig. A1(a). Various baffle designs used in vacuum friction and wear apparatus.

Fig. A1(b), (c), (d). Continued. Various baffle designs used in vacuum friction and wear apparatus.

Fig. A1(e), (f), (g). Continued. Various baffle designs used in vacuum friction and wear apparatus.

Fig. A1(h), (i), (j). Concluded. Various baffle designs used in vacuum friction and wear apparatus.

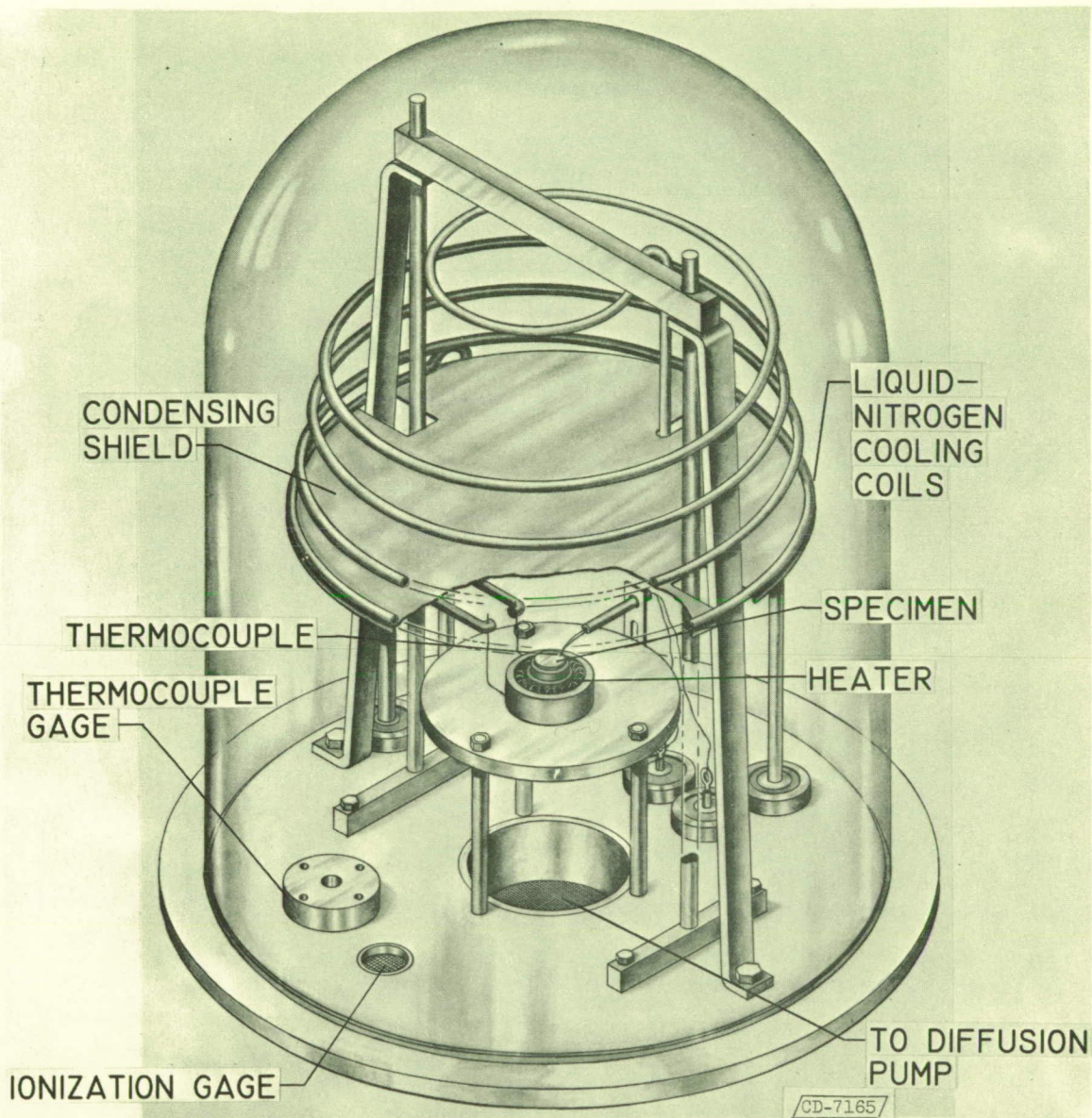


Fig. 1 (a). - Vacuum apparatus for bulk evaporation studies.

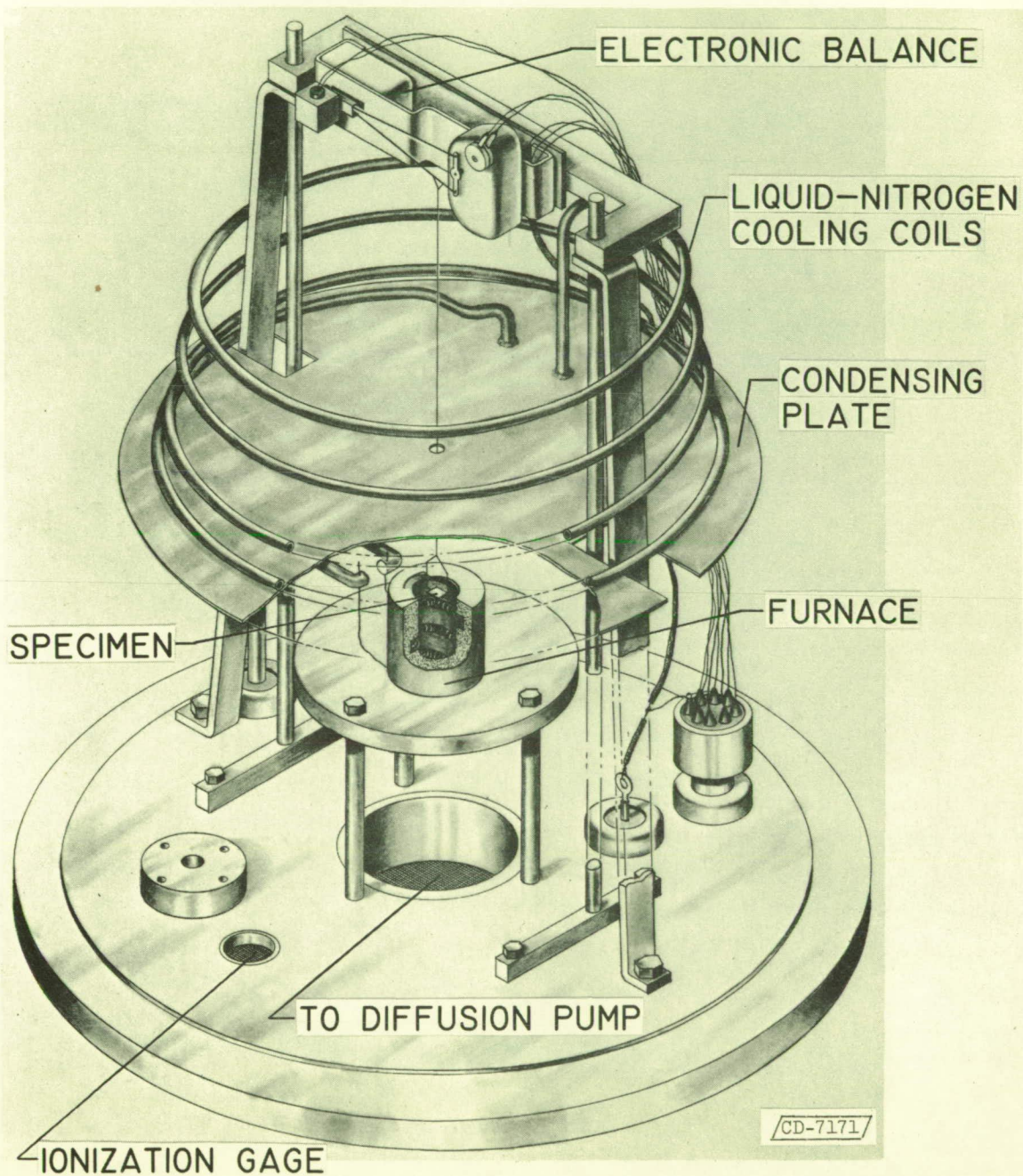


Fig. 1 (b). - Vacuum evaporation apparatus with balance for continuous rate measurements.

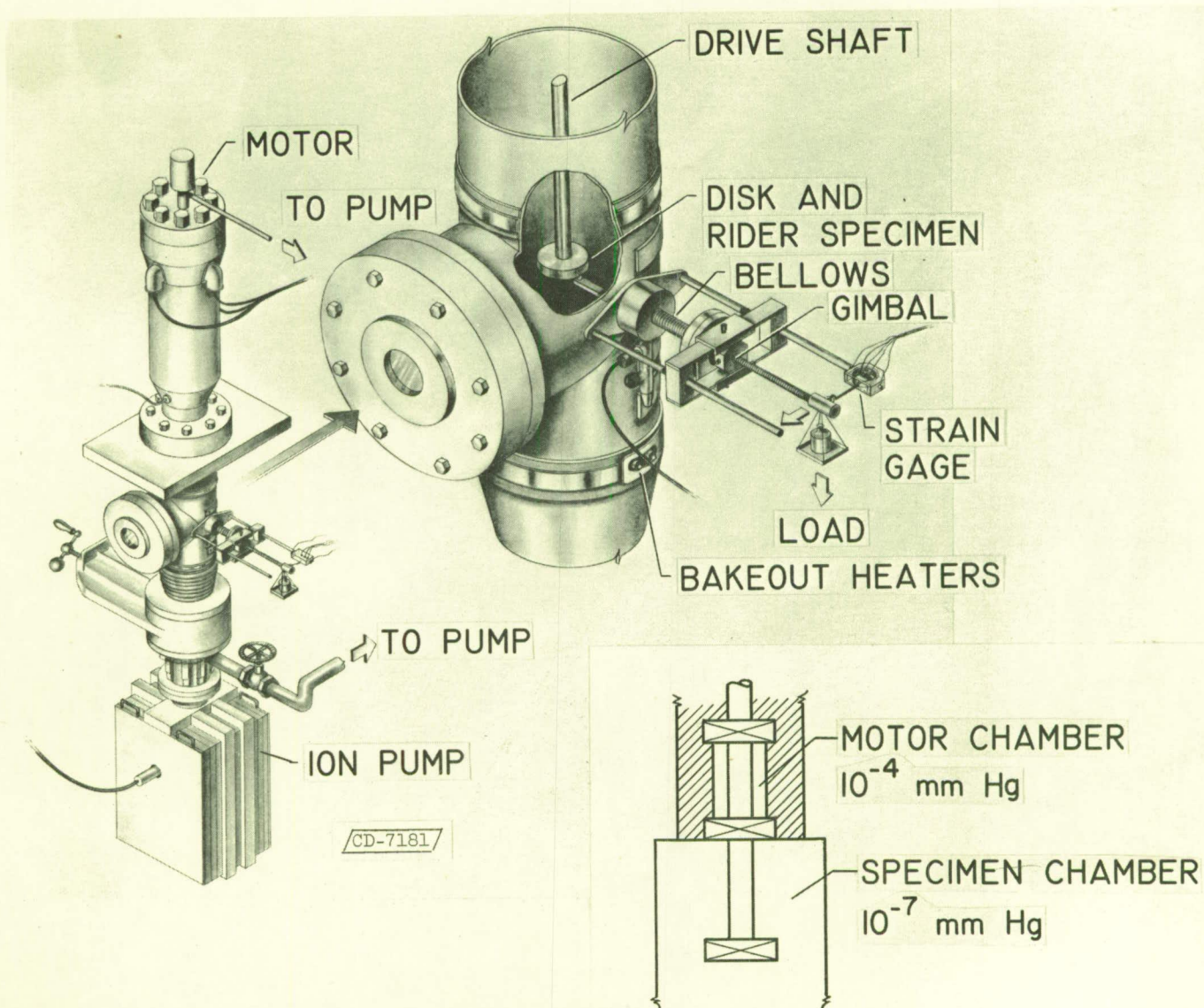


Fig. 1(c). Vacuum friction and wear apparatus.

A - EVAPORATION RATE LESS THAN 1.0×10^{-10} g/cm²/SEC

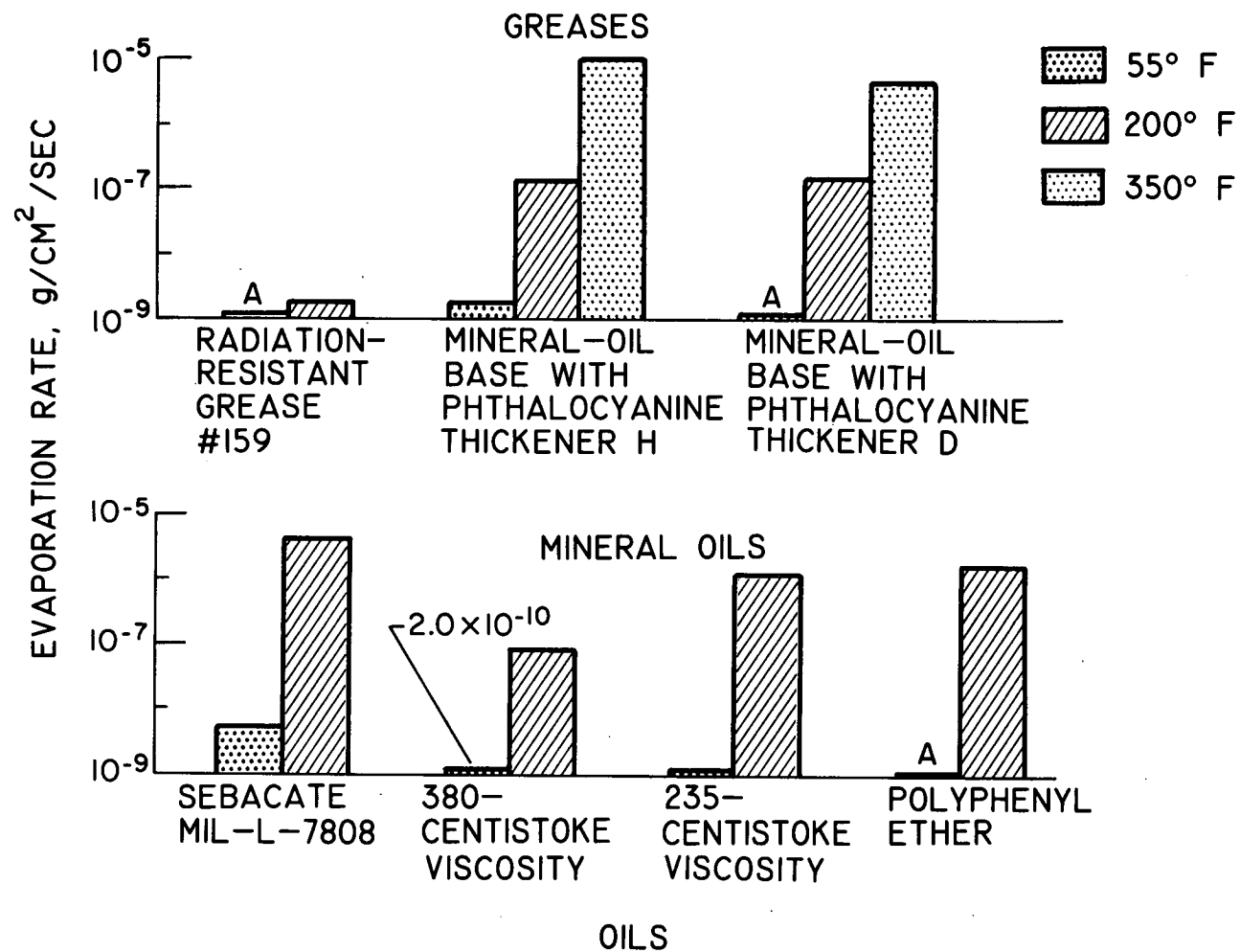


Fig. 2. Evaporation rates for various oils and greases in vacuum. Ambient pressure, 8.0×10^{-7} to 2.0×10^{-6} mm Hg.

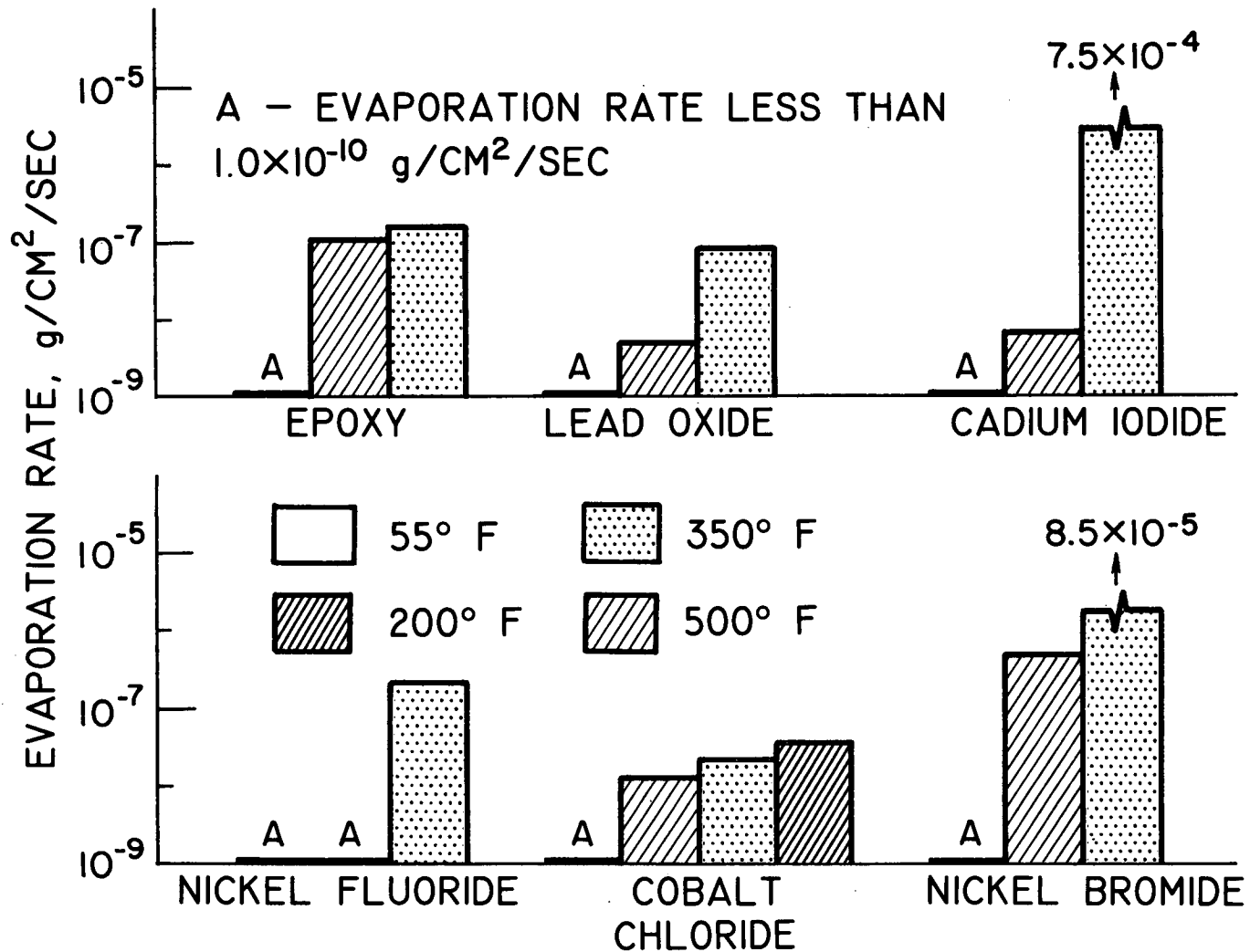


Fig. 3. Evaporation rates for possible coating constituents in vacuum. Ambient pressure, 8.0×10^{-7} to 2.0×10^{-6} mm Hg.

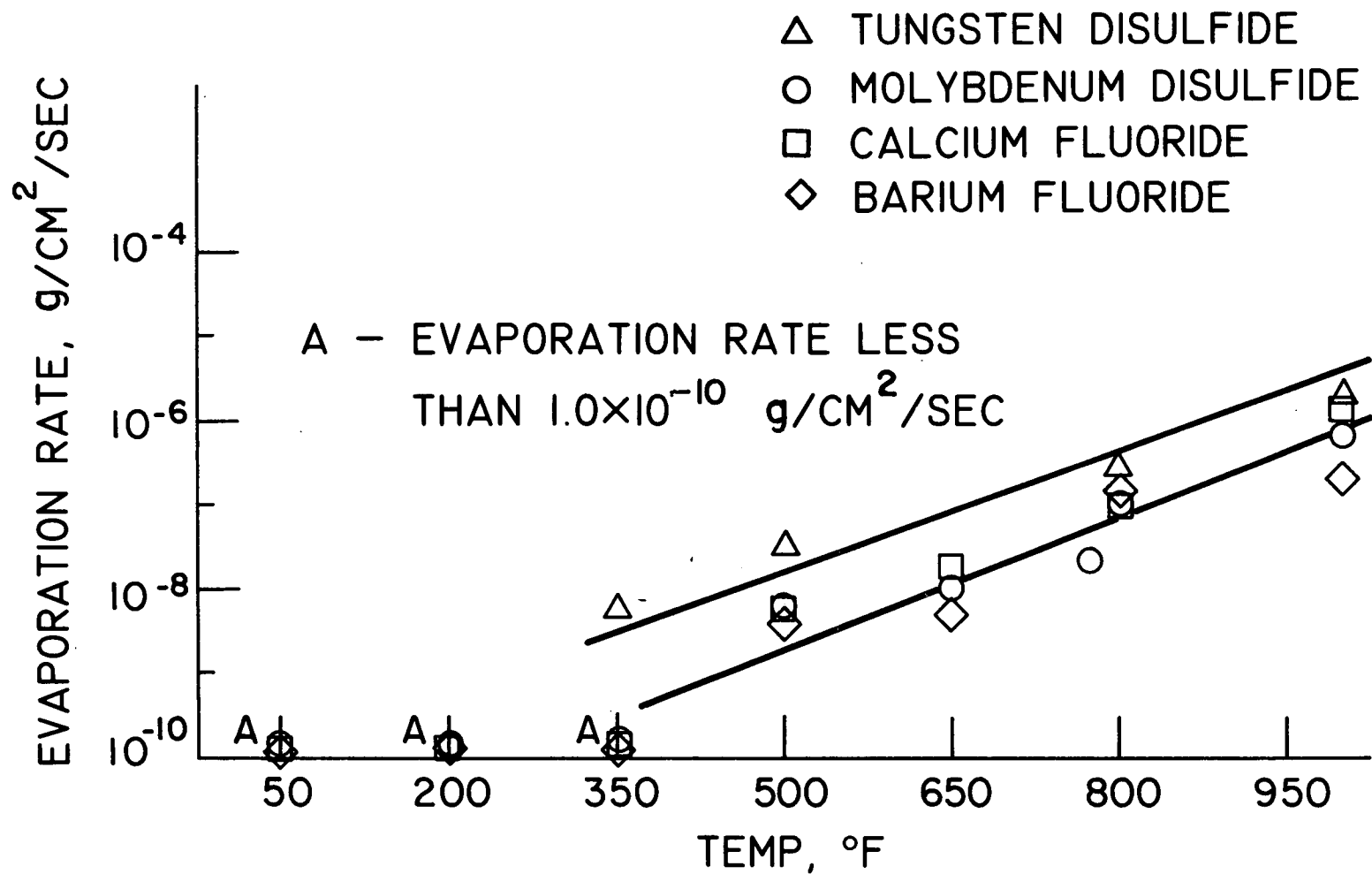


Fig. 4. Evaporation rates for various inorganic compounds in vacuum. Ambient pressure, 8.0×10^{-7} to 2.0×10^{-6} mm Hg.

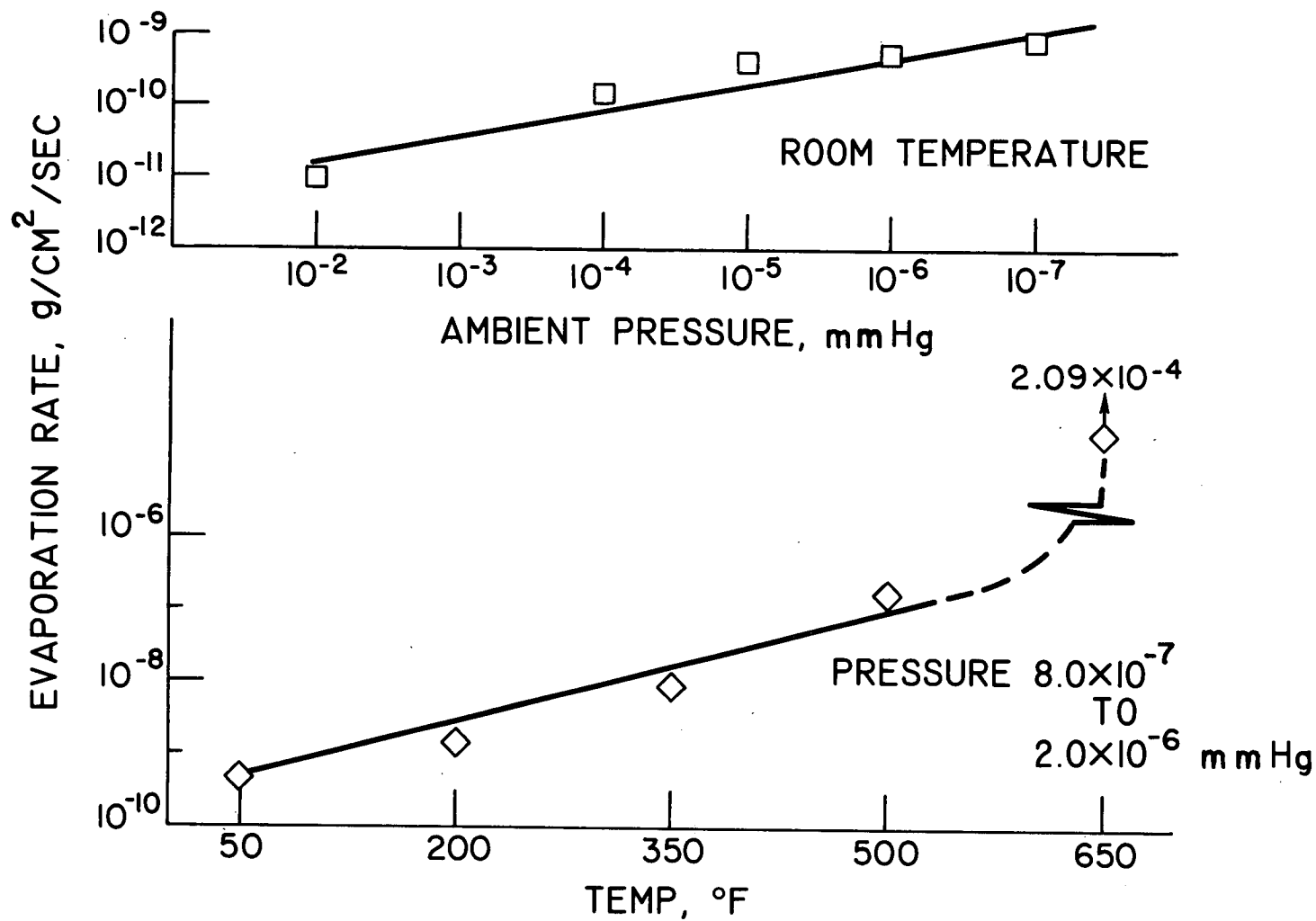


Fig. 5. Evaporation rate of PTFE at various temperatures and pressures.

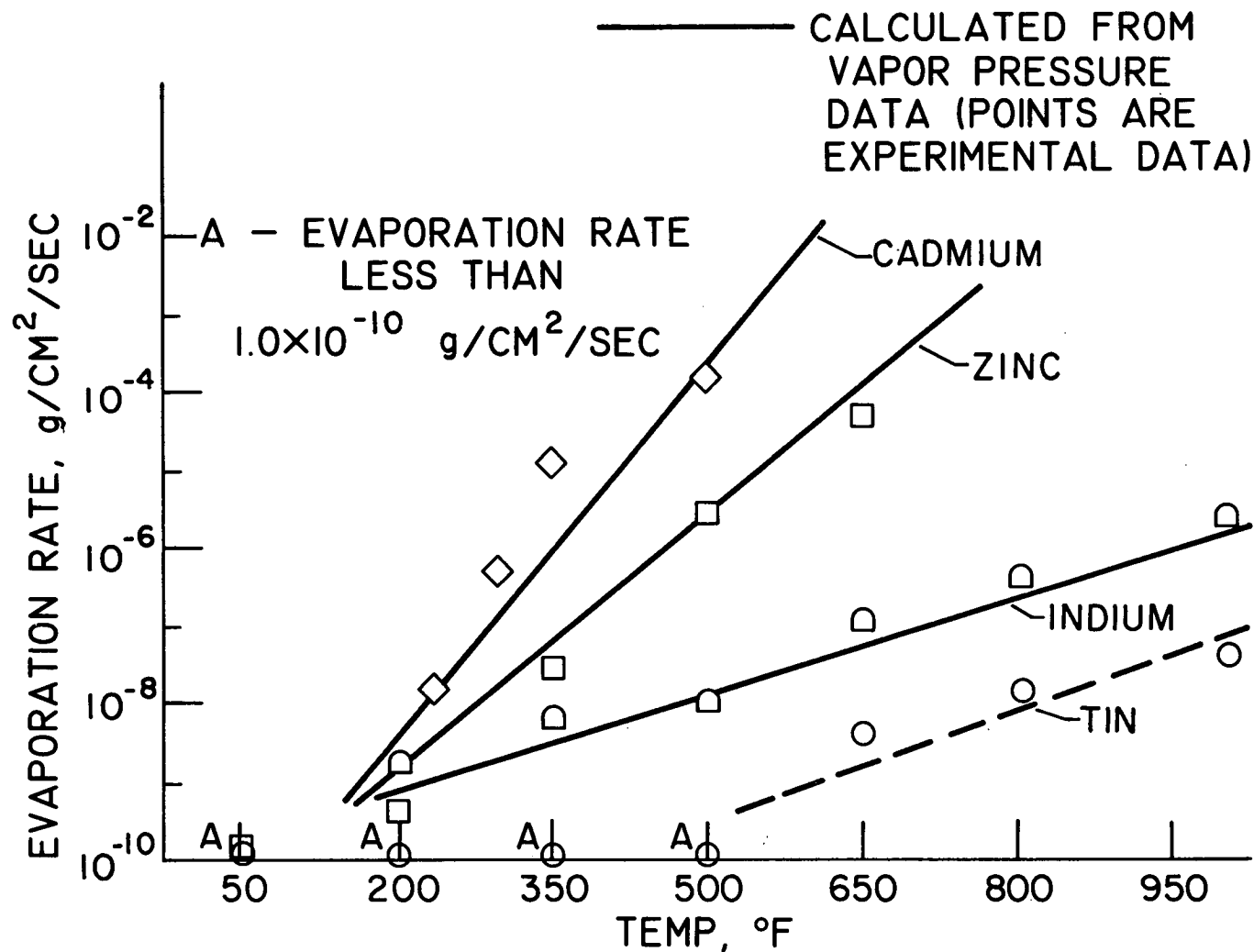


Fig. 6. Evaporation rates for various metals in vacuum. Ambient pressure, 8.0×10^{-7} to 2.0×10^{-6} mm Hg.

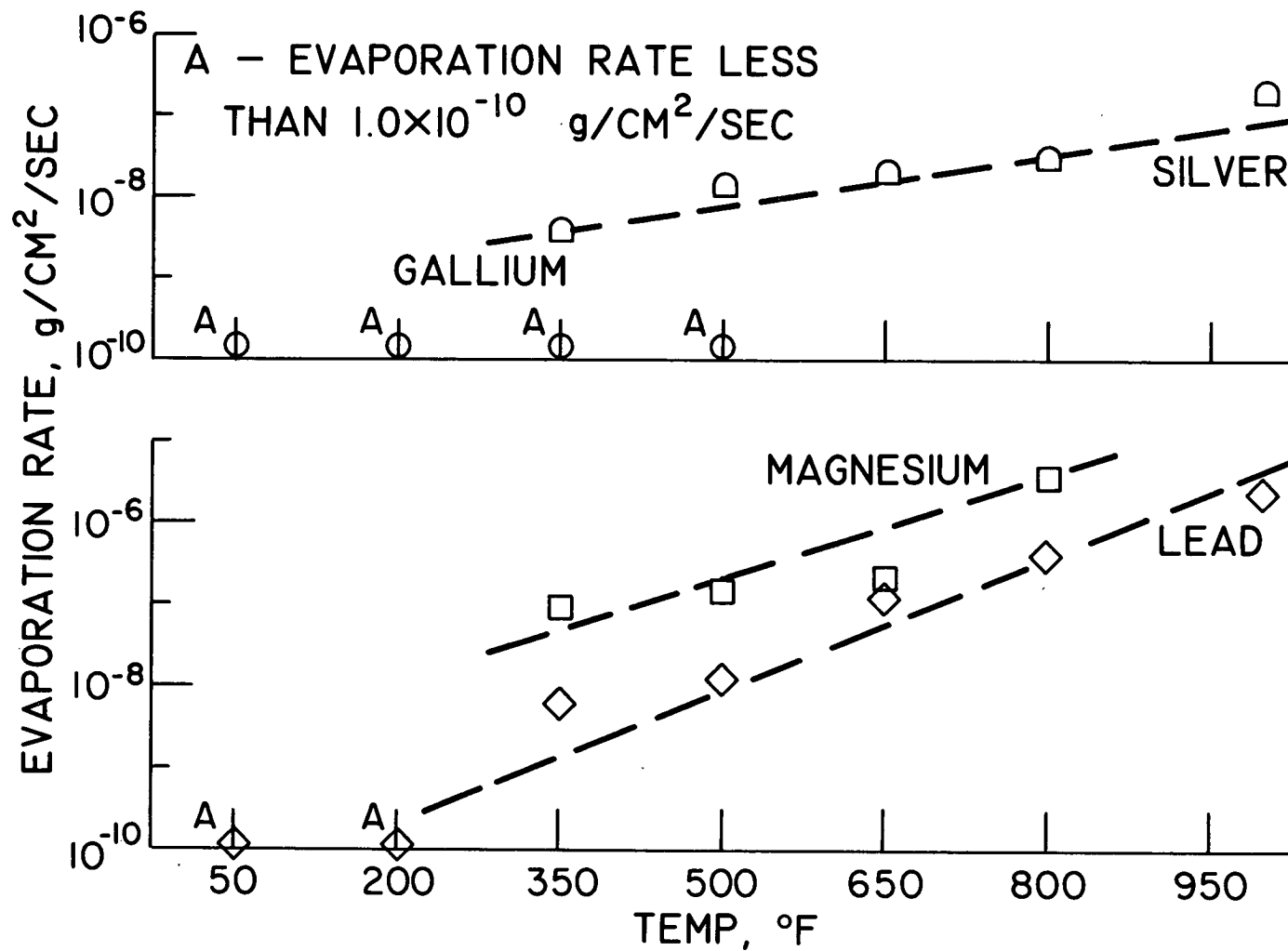


Fig. 7. Evaporation rates for various metals in vacuum. Ambient pressure, 8.0×10^{-7} to 2.0×10^{-6} mm Hg.

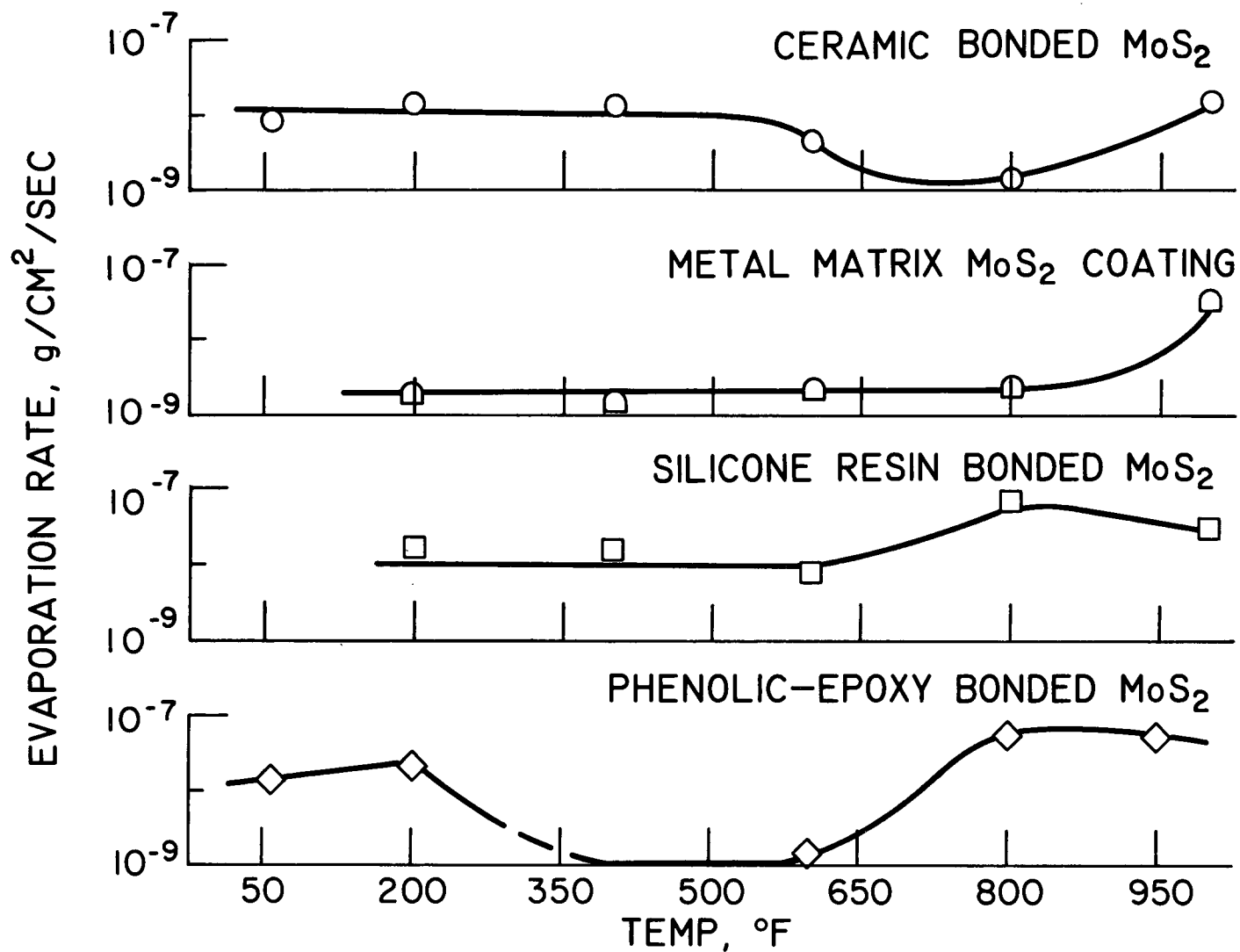


Fig. 8. Evaporation rate of various MoS_2 coatings in vacuum. Ambient pressure, $1.0 - 2.0 \times 10^{-6}$ mm Hg; 0.001-in. MoS_2 coating on Ni-Cr alloy.

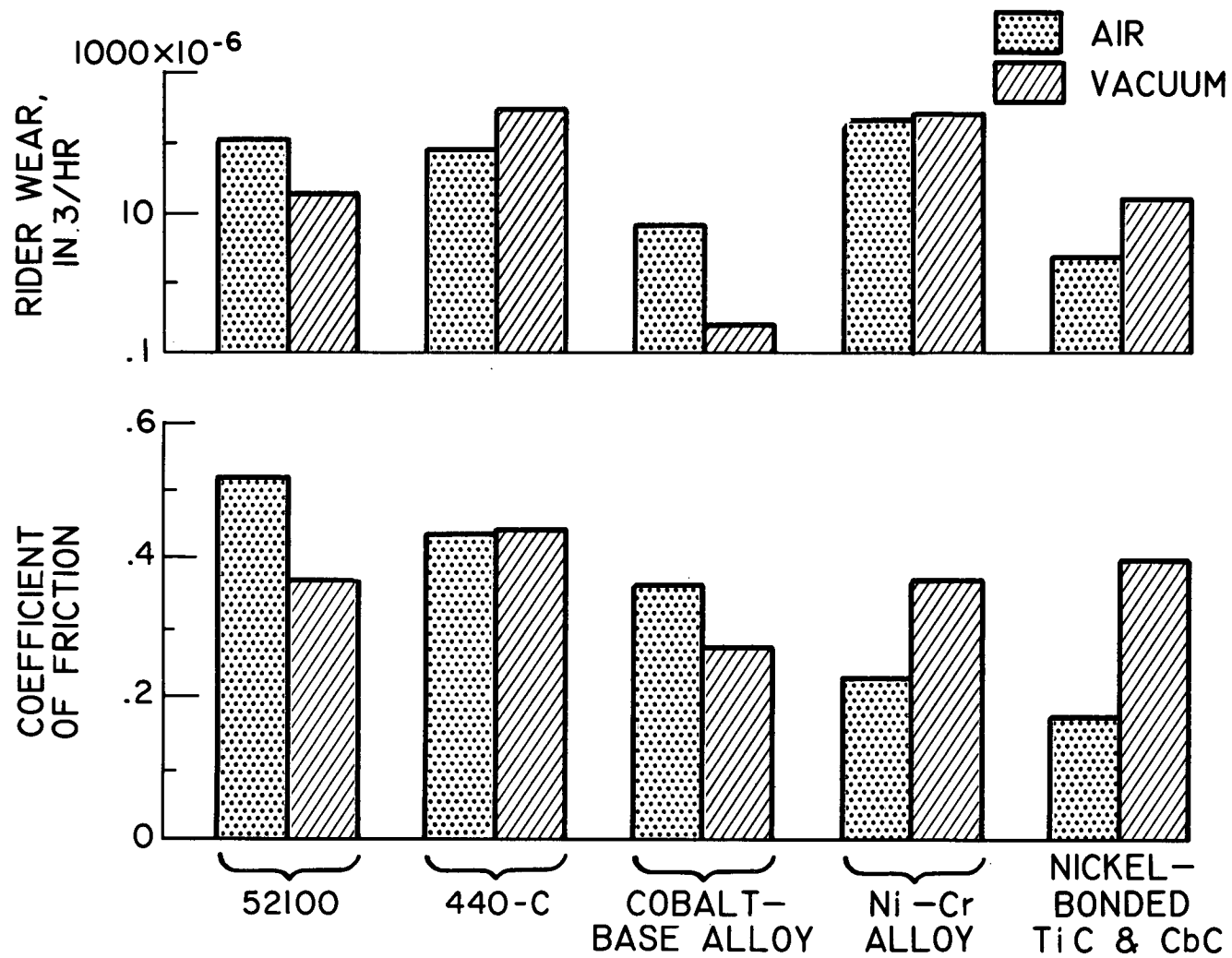


Fig. 9. Friction and wear of various alloys in air and in vacuum. Vacuum, 6.5×10^{-7} to 2.0×10^{-6} mm Hg; sliding velocity, 390 ft/min; load, 1000 g; duration of run, 1 hr.

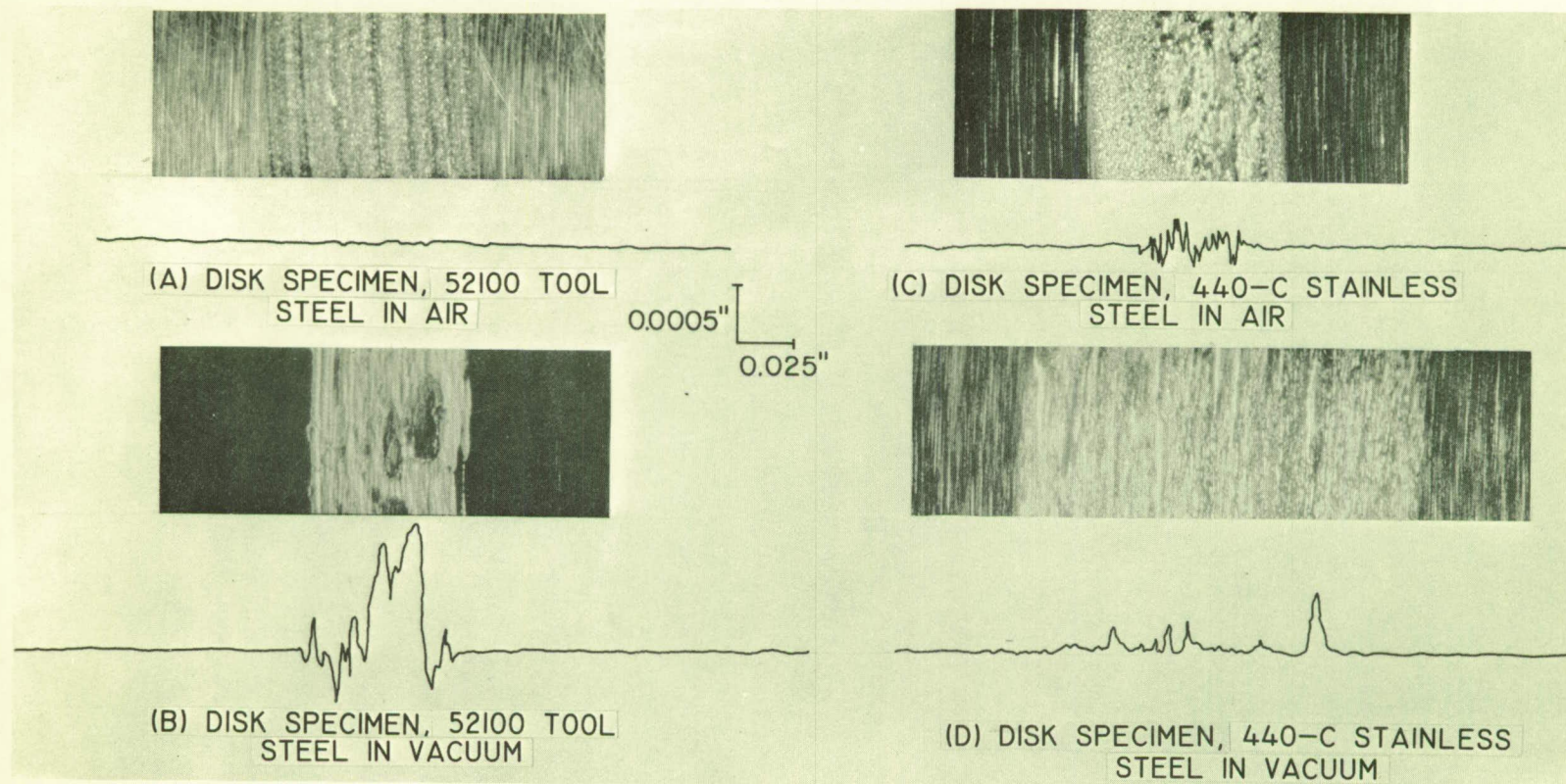


Fig. 10. Photomicrographs and surface profile tracings of wear areas on disk specimens. Load, 1000 g; sliding velocity, 390 ft/min; duration of run, 1 hr. X20.

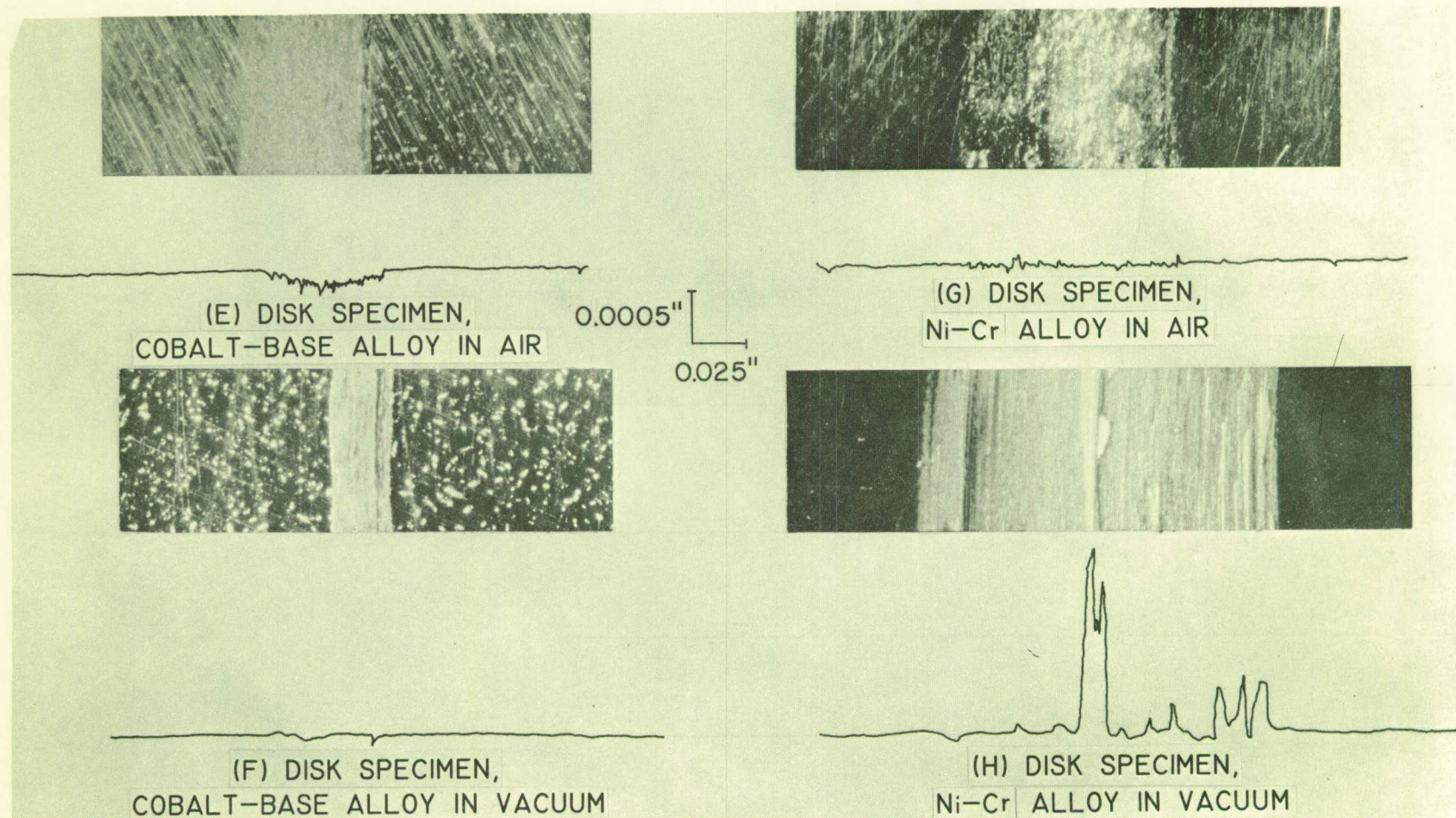


Fig. 10. Continued. Photomicrographs and surface profile tracings of wear areas on disk specimens.
Load, 1000 g; sliding velocity, 390 ft/min; duration of run, 1 hr X20.

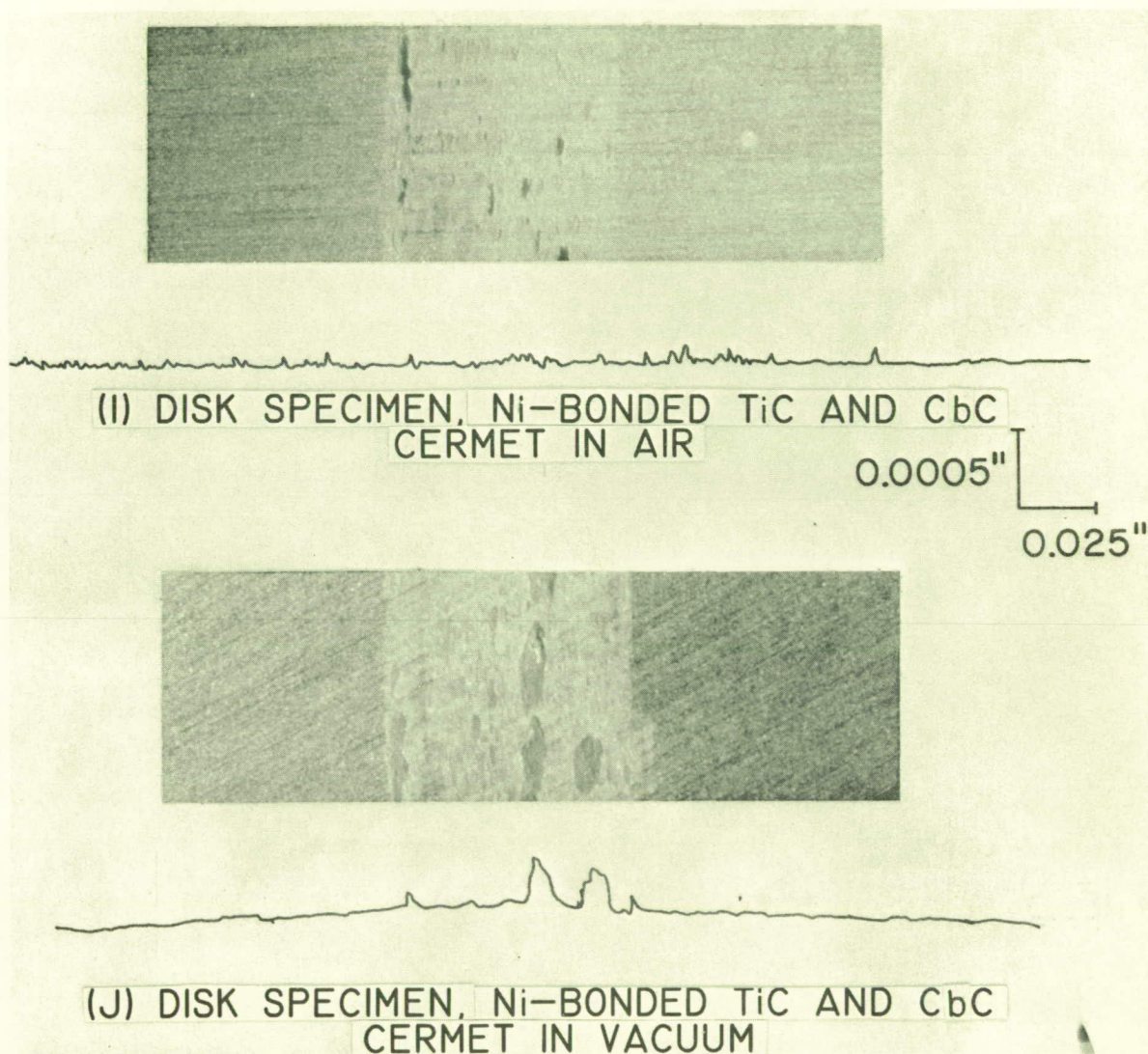


Fig. 10. Concluded. Photomicrographs and surface profile tracings of wear areas on disk specimens. Load, 1000 g; sliding velocity, 390 ft/min; duration of run 1 hr X20.

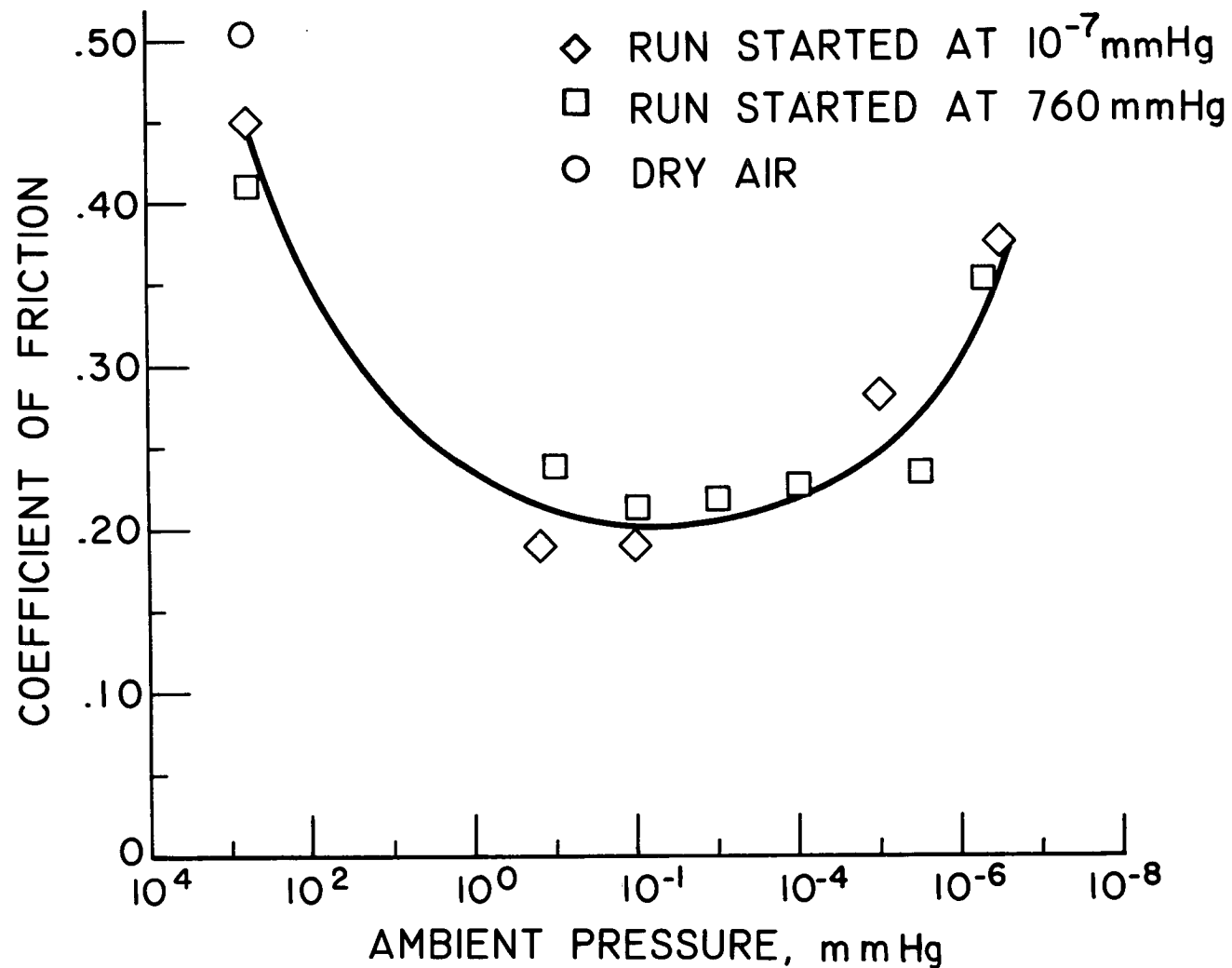


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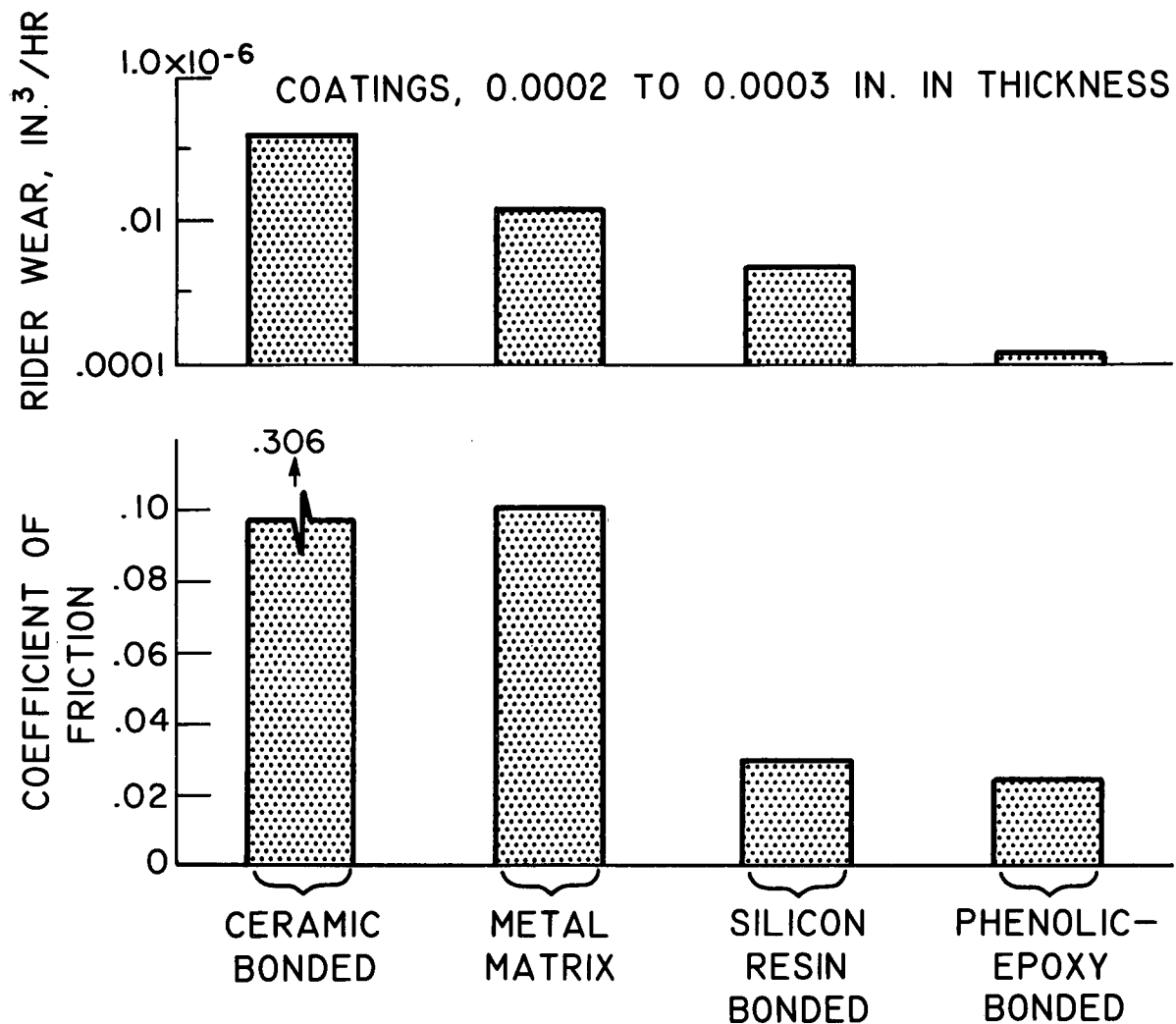


Fig. 12. Friction and wear of 440-C on 440-C with various MoS₂ films. Ambient pressure, 8.0×10^{-7} to 2.0×10^{-6} mm Hg; sliding velocity, 390 ft/min; load, 1000 g; duration of run, 1 hr.

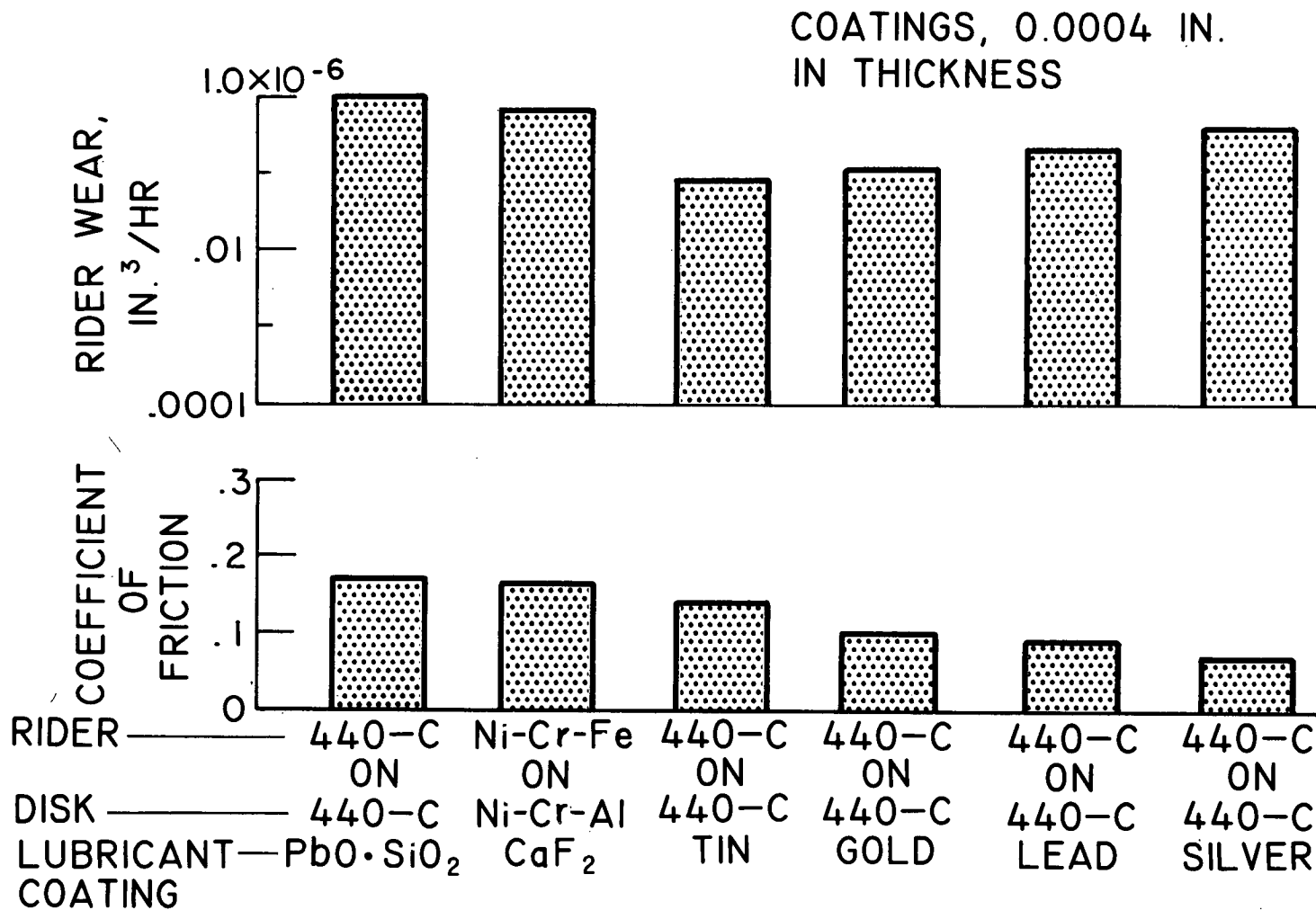


Fig. 13. Friction and wear of alloys with various coatings in vacuum. Ambient pressure, 8.0×10^{-7} to 2.0×10^{-6} mm Hg; sliding velocity, 390 ft/min; load, 1000 g; duration of run, 1 hr.

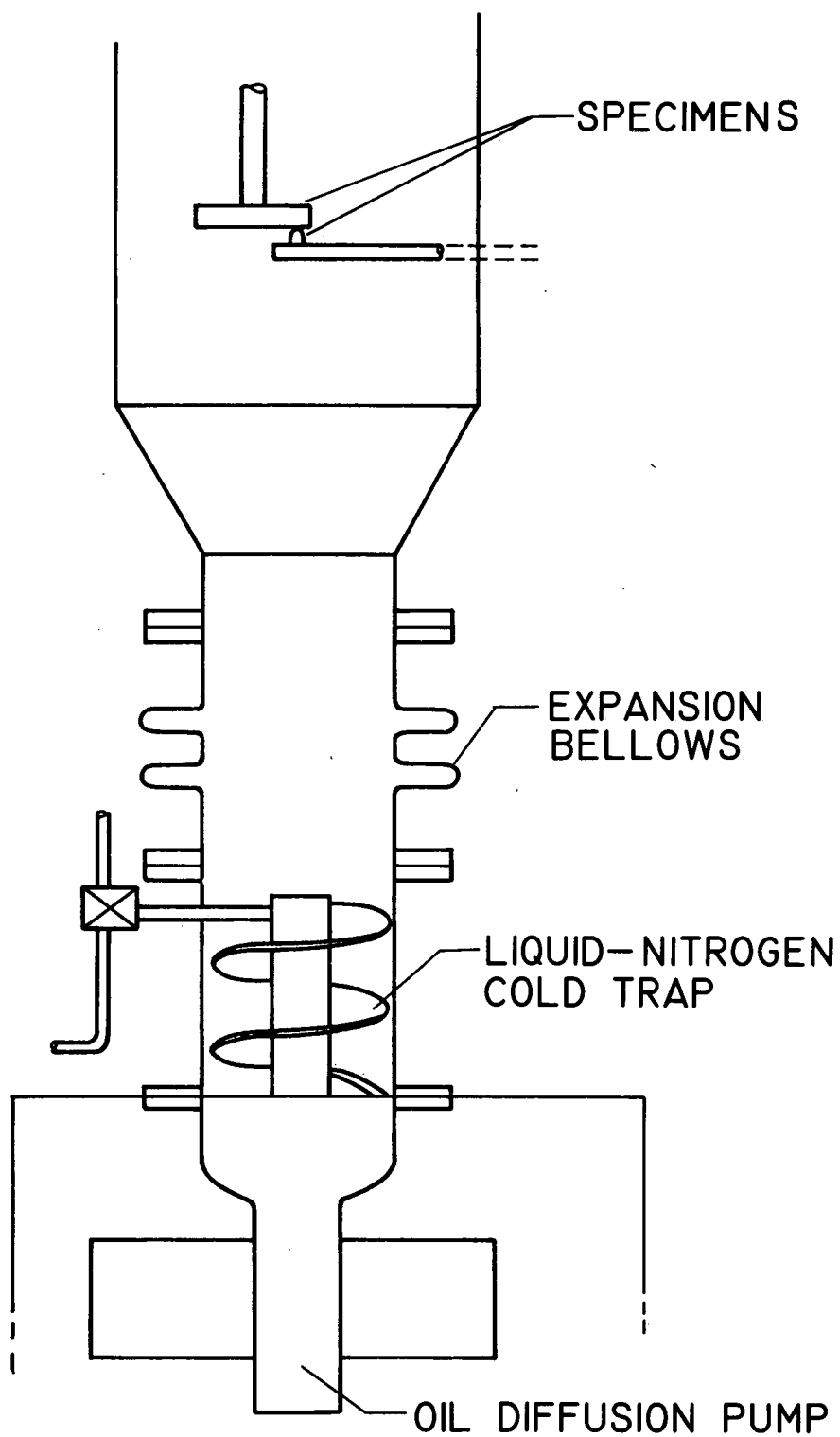


Fig. A1(a). Various baffle designs used in vacuum friction and wear apparatus.

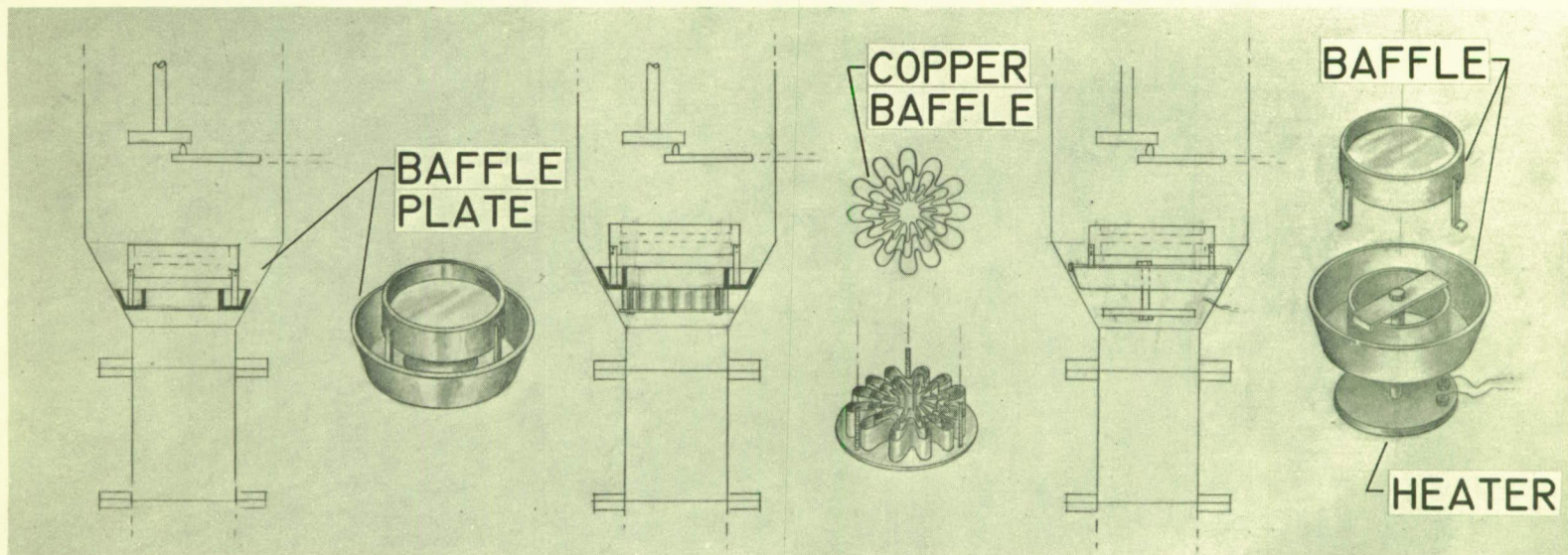


Fig. A1(b), (c), (d). Continued. Various baffle designs used in vacuum friction and wear apparatus.

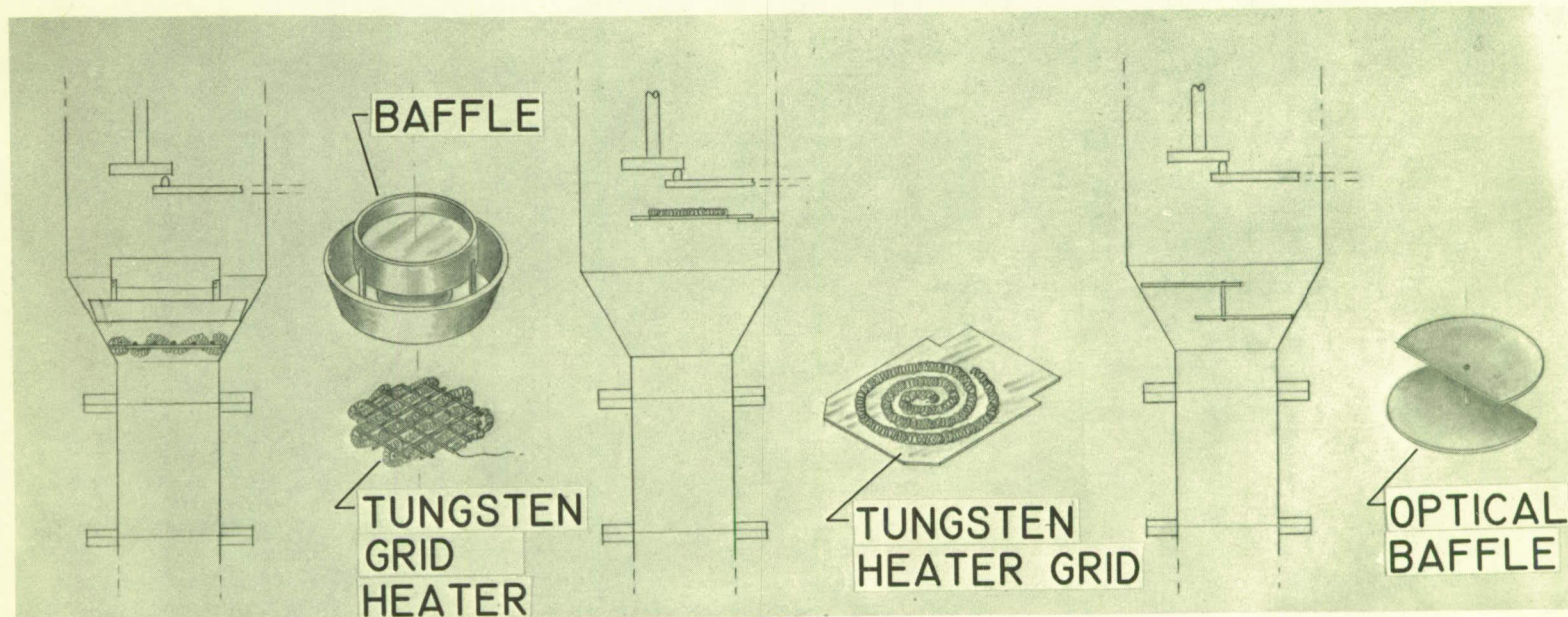


Fig. Al(e), (f), (g). Continued. Various baffle designs used in vacuum friction and wear apparatus.

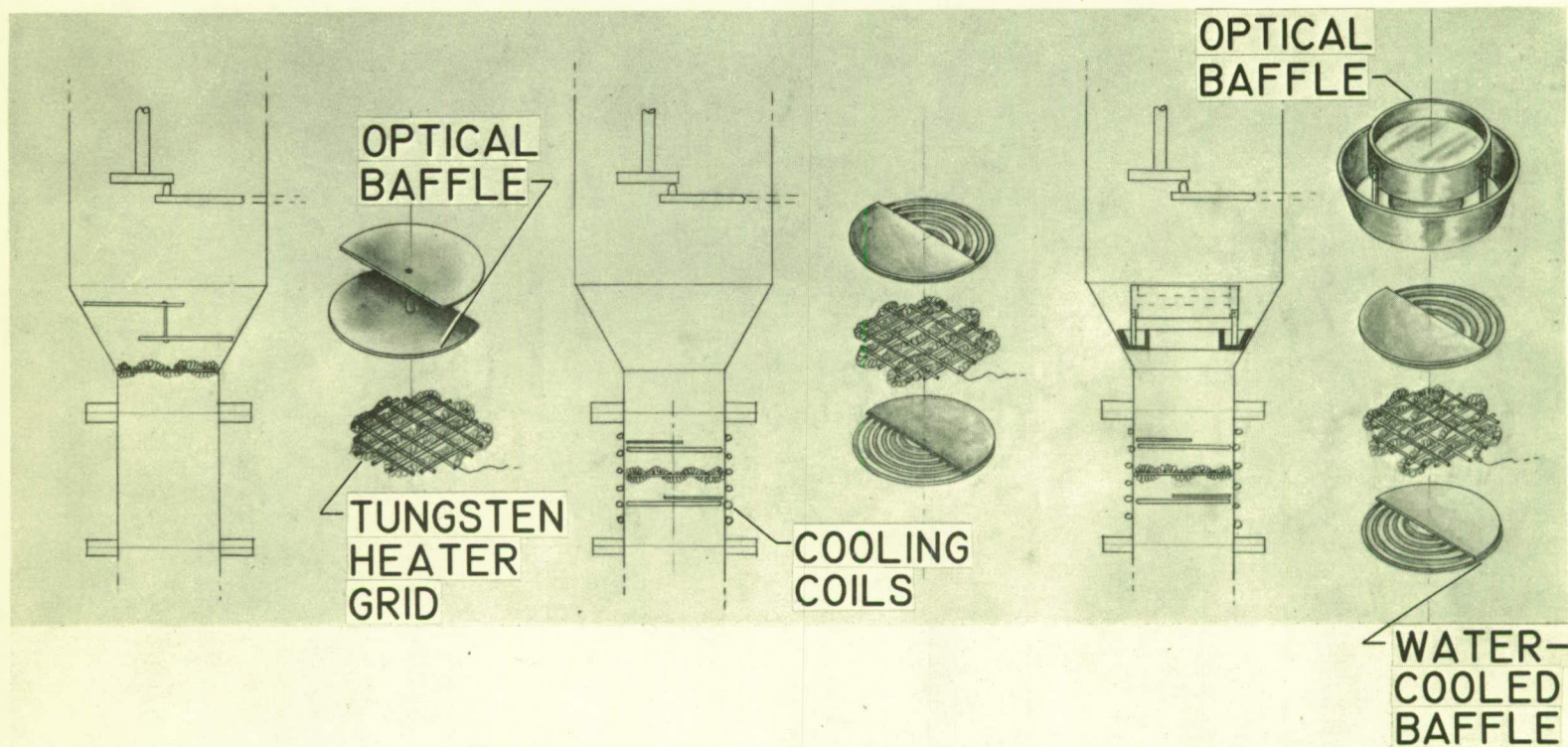


Fig. A1(h), (i), (j). Concluded. Various baffle designs used in vacuum friction and wear apparatus.

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